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ACIDIC PRECIPITATION IN ONTARIO STUDY (APIOS)

PROCEDURES MANUAL
TERRESTRIAL EFFECTS

ARB-93-86-PHYTO

February, 1986

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Ministry
of the
Environment
Ontario

Dr. David Balsillie, Director
Air Resources Branch

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PROCEDURES MANUAL
TERRESTRIAL EFFECTS

ACIDIC PRECIPITATION IN ONTARIO STUDY
(APIOS)

Editor: A. Neary
Dorset Research Centre

ARB-93-86-PHYTO

TECHNICAL SUBCOMMITTEE
TERRESTRIAL EFFECTS WORKING GROUP
ONTARIO MINISTRY OF THE ENVIRONMENT

February, 1986

PREFACE

This manual briefly outlines the procedures followed in the terrestrial effects program of the Acidic Precipitation in Ontario Study (APIOS). It is the second edition of the original version, issued in 1983. The purpose of the manual is to ensure a standardized approach to terrestrial studies carried out by the Ontario Ministry of the Environment throughout the province. It also serves as a source of information to other agencies which may be interested to learn of our study methods. More detailed manuals have been prepared for individual projects in the study.

The manual comprises five parts: vegetation studies, soil studies, biogeochemical studies, forest productivity and decline studies and laboratory analysis. The methods described are those in use at the time the edition of the manual was prepared. The Terrestrial Effects Working Group and the Biogeochemistry Working Group periodically reviews the manual and revises it as required. Many of the methods outlined are those in regular use by the Ministry or generally recognized as acceptable by the scientific community. Procedures representing novel approaches (eg. parts of the biogeochemical program) have been reviewed and approved by scientists within and outside the Ministry.

Principal contributors to the manual were: W. Gizyn and A. Kuja, Air Resources Branch (vegetation studies); D. Dimma, Air Resources Branch (soil studies); W. McIlveen, Northeastern Region (biogeochemical studies); and A. Neary, Laboratory Services Branch (laboratory analysis).

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A. VEGETATION STUDIES

BASELINE SAMPLING

Objective

There are many potential effects of acidic precipitation on terrestrial ecosystems, some of which may lead to changes in foliage chemistry. These effects may occur through direct mechanisms, such as deposition of sulphur and nitrogen, or through accelerated leaching of foliage. Indirect mechanisms, such as changes in nitrogen transformations in soil, leaching of soil bases, increased availability of metal elements, or reduced availability of phosphorus to plants, may also play a role.

The purpose of baseline sampling is to establish the current status for nutrients and selected trace elements in Ontario forest vegetation. This data base will enable future changes caused by acidic precipitation or other long-range transport pollutants to be detected.

Site Selection and Description

To relate changes in foliage chemistry to pollutant deposition, the location of survey sites are as close as possible to APIOS deposition monitoring sites.

Each sampling site is in a secure location, preferably on publicly-owned land. The area is forested, and free of major recent disturbances such as fire, clear cutting, or cultivation. Some assurance that disturbance will not likely occur in the future is also required. Forested lands within provincial parks or conservation areas often meet such criteria.

The site contains as many as possible of the following tree species: sugar maple (Acer saccharum), white birch (Betula papyrifera), trembling aspen (Populus tremuloides), white spruce (Picea glauca) and eastern white pine (Pinus strobus). These species have been selected as common denominator baseline forest trees. However, because of the wide range in climatic and soil conditions across the province, all species are not available at all sites.

Baseline sampling sites are located away from local sources of atmospheric contaminants. Sample trees are at least 100 m from gravel roads, at least 50 m from paved roads, and at least 50 m from the edge of the forest canopy.

Sites are identified by UTM coordinates and a sketch map is prepared to show sampling location in relation to permanent local landmarks. Sufficient detail is included to permit future site relocation. Photographs of the site and its access are taken. To date, 76 sites throughout Ontario have been sampled.

Sampling Procedure

Triplicate representatives of each common denominator species are selected as sample trees. These trees must be healthy, and free of signs of bole damage or crown dieback. A diagram is prepared to indicate the relative distances and compass bearings between the individual sample trees. Plastic tags are prepared showing the program, site number, tree species and replicate number. For example, a tag reading "APIOS - S7 - PG 3" would represent the APIOS program, site #7, Picea glauca, replicate 3. These tags are attached to the stem of the tree with a stainless steel nail, which is only partially driven in to permit stem diameter growth. Alternatively, numbered tags may be used.

Branches are cut from the middle, or lower-middle portion of the crown. To increase the possibility of obtaining a representative sample, the lowest branch is avoided, even though it is the most accessible. Pruning poles of sufficient strength and length are recommended as sampling devices to reach such branches, which are often more than 15 m above the ground. To avoid contamination, the falling branch is not permitted to contact the mineral soil. Foliage-bearing twigs are cut from the branch and placed in plastic bags. Plastic gloves are worn to prevent sample contamination. Appropriate sample numbers are attached to the sample bags.

Sample Preparation and Submission for Analysis

Samples are refrigerated as soon as they are brought to the processing laboratory. The leaves, including petiole, are first removed from the twigs. Damaged or infected leaves are discarded. For white pine, the current year's foliage is separated from older foliage and the two samples are processed separately. For white spruce, the current year's foliage is separated from 1-year-old foliage and the two samples are processed separately. Foliage more than 1 year old is discarded.

Sorted samples are placed in kraft paper bags and dried for 30 hours at 80°C in a forced draft oven. Dried tissue is ground in a stainless steel Wiley mill to pass through a screen with 1 mm mesh and is collected in a glass jar with a plastic or pulp-lined lid.

Baseline samples are submitted to the Ministry's Toronto laboratory for the following analyses: total sulphur, nitrogen, phosphorus, potassium, chloride, calcium, magnesium, sodium, aluminum, manganese, iron, cadmium, copper, molybdenum, nickel, lead and zinc. The LIS (Laboratory Information System) test group for these parameters is TEAPS 5 and the individual LIS test names are given in Section E. Laboratory Analysis.

Variability Study

In 1982, a special site was established which is used to provide a measure of the variability which can be expected when using the vegetation sampling protocols outlined for the baseline survey. The site selection criteria, sample processing and analytical procedures were identical to those used in the baseline survey, however, sampling was more intensive. Six trees, representing a range of diameter classes, were sampled from each of the following species: white pine, sugar maple and white birch. Sampling was done in an area with an approximately 25 km radius. Samples were collected in late June and Mid-August at two heights and from two sides of each tree canopy.

SIMULATED ACID RAIN STUDIES

Objective

The objective of these studies is to document the effects of acidic precipitation on vegetation under controlled conditions using indoor rain chambers and outdoor rain exclusion canopies. Experiments are carried out to determine: threshold doses causing injury symptoms; biochemical and physiological effects; relative sensitivity of agricultural crops and forest tree species; growth and yield response of plants to different rain acidities; effects on seedling establishment; and effects caused by interactions with gaseous pollutants and with plant pathogens.

Program Management

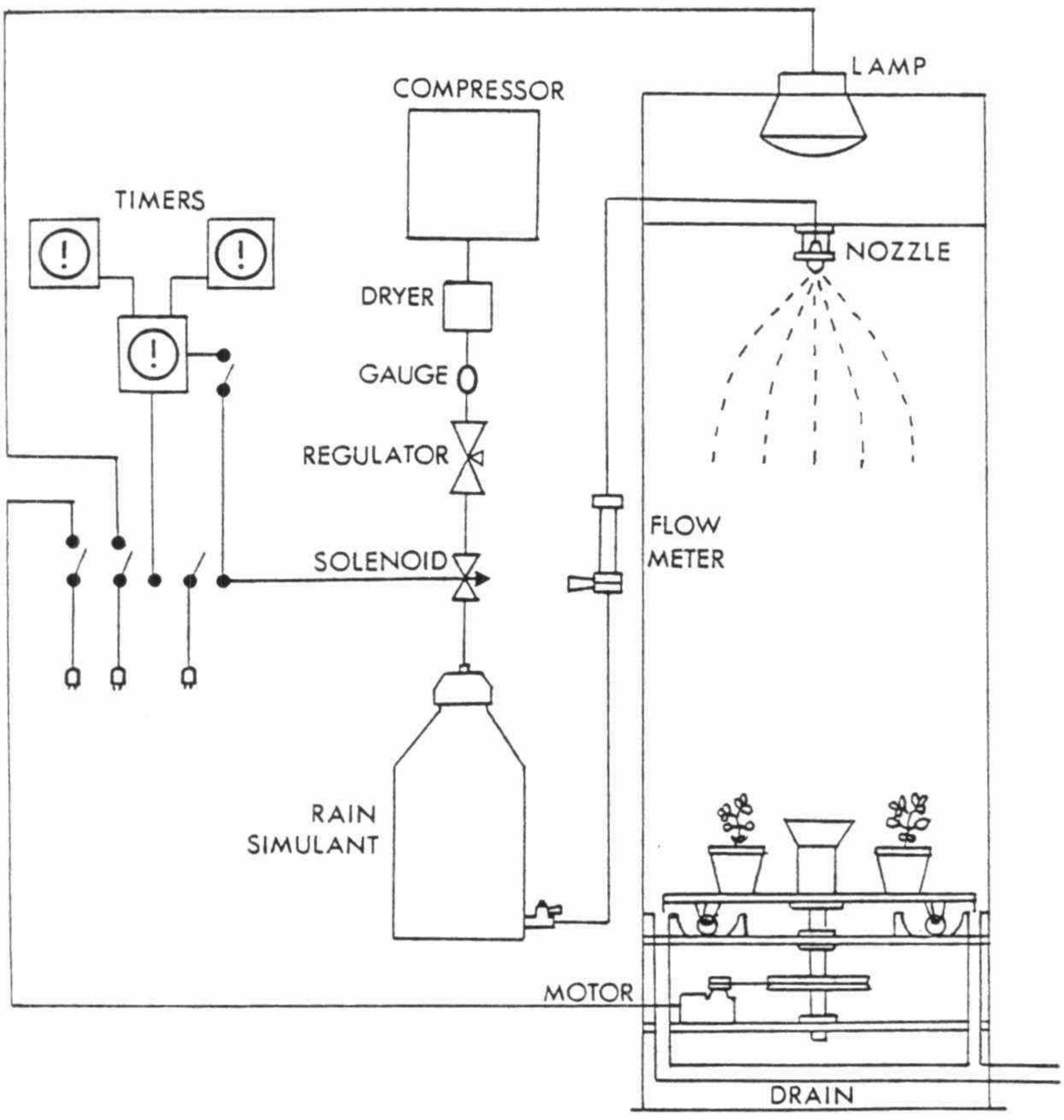
Simulated acid rain studies in the APIOS progam are carried out at a controlled environment laboratory at Brampton, Ontario, by the Ministry's Phytotoxicology Section, Air Resources Branch. All proposed experiments are first reviewed by the Technical Subcommittee, Terrestrial Effects Working Group, which sets priorities and ensures that the studies comply with established objectives.

Equipment

Simulation Chambers

Four chambers, permitting the simultaneous application of four different simulated rain solutions, are available for experimental work. The chambers are housed in a single framework 7.3 m high, 4.9 m wide, and 1.2 m deep, constructed of aluminum angle. Each chamber floor is 1.2 by 1.2 m. A steel platform along the top of the structure provides access to nozzles and light fixtures. Figure 1 is a schematic diagram of a chamber and ancillary control apparatus.

The chamber walls consist of an outer layer of extruded styrofoam and a water-resistant inner layer of white, laminated, plastic. Chamber walls are secured to the aluminum frame with



Note: The system consists of four chambers each with identical components. For clarity only one is shown here.

Figure 1. Schematic Diagram of Indoor Rain Simulation Chambers

stainless steel bolts. Seams and joints of inner walls are sealed with silicone to prevent leakage. A clear, plexiglass door, 1.14 m high and 0.8 m wide, provides access to each chamber at a point just above the turntable. Chamber floors are constructed of fibreglass-coated, 20-mm plywood, and drained with ABS plastic piping, 5 cm in diameter.

A turntable, which permits uniform distribution of simulated rain, is located on the floor of each chamber. Each turntable is 1.1 m in diameter and is constructed of 20-mm plywood. It is driven by a central steel shaft connected to a 1/16 hp electric motor beneath the chamber floor. The turntables rotate at 4 r.p.m. The weight of potted plants is supported by four pivoting caster wheels on the underside of the turntable. The turntable in each chamber can accommodate up to 80, 10-cm pots; 36, 16-cm pots; or 20, 22-cm pots. However, about half this number is more manageable so that test plants do not touch each other. Pots are arranged in a circle and are equidistant from the centre of the turntable. Each turntable is fitted with a skirted PVC cover, which is removable for cleaning or maintenance.

To achieve terminal velocity, droplets of the simulated rain solution are dispersed from a nozzle 5.33 m above the centre of each turntable. The nozzles are fabricated from a stainless steel body, and a PVC nipple which produce droplets 1 mm in diameter, within the size range of natural raindrops. The droplets are dispersed under low pressure (2 - 3 psi) from 28 orifices. Each nozzle is attached to the centre of a plexiglass levelling plate, which can be adjusted to produce the best "rain" distribution on the turntable below. Nozzles are connected with 6-mm O.D. polyethylene tubing and compression fittings to the rain application system at the base of the chambers.

To simulate light conditions during natural rainfall, a fixture containing a 1000-watt, metal halide lamp is mounted above each chamber nozzle. To obtain optimum light at the turntable, reflective, heavy-gauge, aluminum foil is placed around each fixture.

The tops of the four chambers are presently uncovered. They will be sealed when plans for temperature and humidity control are implemented.

Rain Application System

Simulated rain solutions are stored in eight, stainless-steel, 20-litre liquid storage tanks, located near control panels which govern the application of the solutions in the four chambers. During treatment applications, the pressure in each tank is maintained at 15 psi with a regulator mounted in the line from the compressor supplying filtered, dry air. Three electric timers may be programmed to supply rain simulants at a continuous rate up to 6 h, or intermittently for intervals up to 12 min each within any given period of up to 6 h. The timers activate solenoid valves which control the flow of solutions from the tanks to the dispersal nozzles. Flow rates are controlled by rotometers. Between 0.5 and 2.5 cm of simulated rain per hour is applied to test plants with this system when operated in the continuous mode. A rate of 1.0 cm per hour gives maximum coverage of the turntable.

Simulated Rain Solutions

A treatment range of pH 2.6 to 5.6 is used for simulation experiments, based on the pH of "normal" rain and the current pH range of rainfall in Ontario. A similar range is used in laboratories in the United States. Eight pH levels are presently used in the Brampton chambers to determine threshold effects and to construct dose-response curves: 2.6, 3.0, 3.4, 3.8, 4.2, 4.6, 5.0 and 5.6.

The composition of stock solutions prepared for simulation experiments is based on the chemistry of rainfall monitored in Ontario. A working background ion stock solution (pH 5.6) has the following composition: Na^+ (0.10 mg/ℓ), K^+ (0.064 mg/ℓ), Mg^{++} (0.10 mg/ℓ), Ca^{++} (0.50 mg/ℓ), NH_4^+ (0.75 mg/ℓ as N), Cl^- (0.02 mg/ℓ), NO_3^- (1.38 mg/ℓ as N), and $SO_4^=$ (2.77 mg/ℓ). This working stock solution has a SO_4:NO_3 ratio of 2:1 and is used "as is" for the pH 5.6 treatment.

For the remaining seven pH treatments, separate acid working stock solutions are prepared from a H_2SO_4 super stock solution. Dilution of 100 mℓ of each of the acid working stock solutions with 25 litres of working ion stock solution will yield the desired pH for the treatment and maintain a 2:1 SO_4:NO_3 ratio. If future monitoring detects a change in the ratio in ambient rainfall, the ratio in simulant solutions will be adjusted appropriately (Table 1).

All stock solutions are stored in opaque, 1-litre "Nalgene" containers at 4°C, and are equilibrated at room temperature before use. Rain simulants are prepared in 25-litre batches. Water for dilution of stock solutions is provided by a water purifier which uses reverse osmosis and a mixed deionizing resin-bed cartridge. This unit produces 32 litres of ultra-pure water per hour, at a resistivity of 18 megohm. Each batch of simulant must be within 0.1 pH unit of the desired pH. Samples of simulants and deionized water are routinely submitted to the Ministry's Toronto laboratory for analysis of conductivity, pH, Gran acidity, SO_4, NO_3, NH_3, Ca, Na, K, Cl, PO_4, Fe, Pb, Zn, Cu, Ni and Cd. LIS test names are given in Section E. Laboratory Analysis.

Preparation of Test Plants

Soil mixtures are prepared from Ontario Ministry of Agriculture and Food recommendations for food crops. Pot size for each experiment is based on plant size and the duration of the experiment. To avoid toxic effects on hypocotyl development, only the bottom half of each pot has fertilized soil, with the upper half containing unfertilized soil.

In experiments studying the direct effects of acid rain on plant foliage, rain simulants are excluded from the soil by mounding the soil about 2 cm above the pot rim and covering it with thin, plastic sheeting secured by rubber bands. The sheeting is sufficiently loose to allow the soil to "breathe". Two short, cross-shaped slits are cut in the centre of the plastic film through which seeds are sown. Pots covered in this manner are irrigated from below.

Table 1. Chemical composition of simulated acidic rain (SAR) solutions.

pH of SAR	Ion Species Concentration (mg dm^{-3})*			S/N[¶] Ratio
	H^+	$SO_4^=$	NO_3^-	
5.6	0.003	2.769	1.385	1.99:1
5.0	0.010	3.115	1.558	1.99:1
4.6	0.025	3.638	1.820	1.99:1
4.2	0.063	4.954	2.476	2.00:1
3.8	0.158	8.245	4.126	1.99:1
3.4	0.398	16.554	8.268	2.00:1
3.0	1.000	37.399	18.684	2.00:1
2.6	2.512	89.705	44.851	2.00:1

*All simulated rain solutions contained the following ions: ammonium - 0.744 mg dm^{-3}, calcium - 0.500 mg dm^{-3}, magnesium - 0.097 mg dm^{-3}, sodium - 0.100 mg dm^{-3}, potassium - 0.063 mg dm^{-3}, chloride - 0.023 mg dm^{-3}.

[¶] Sulphate to Nitrate Ratio (by Mass).

All agricultural seeds are obtained from registered suppliers and stored at 4°C. Forest tree species are collected as seedlings from natural areas or aquired as transplants from nursery stock. Seed age and viability is recorded. Seeds are usually sown four to a pot and, after germination, thinned to one healthy individual. Pots are normally irrigated by placing them in plastic trays containing 1-2 cm deionized water, except when an experiment calls for all moisture to be supplied from rain treatments.

After treatment, all pots are placed in random positions on greenhouse benches to reduce effects caused by differences in light intensity. Temperature and humidity are controlled and recorded. Incoming air is filtered to remove gaseous and particulate pollutants. Each of the two greenhouses is also equipped with a mechanically-controlled, heavy-gauge, polyethylene curtain to partially control incoming and outgoing radiation through the greenhouse roof. Fluorescent and incandescent lighting, controlled by timers to provide extended day length, is also available. During experiments, light is measured three times daily at three points along each bench where test plants are placed, using an integrating radiometer/ photometer with quantum sensor.

Treatment Applications

All pots are carefully handled in the early seedling stage to avoid damage to fine roots. Pots are arranged in a circle on the turntable and are equidistant from its centre. When the rain treatment has ended, plants are carefully removed from the turntable so that droplets on the foliage are not disturbed. To account for differences between chamber environments or nozzles, rain simulants are rotated among the four chambers so that all chambers receive each treatment during any experiment. Each nozzle system is briefly flushed with deionized water between treatments. Just before and after treatment, temperature and humidity in each chamber are measured with a digital thermohygrometer.

If simulants are the only source of water, treatments are usually applied every second day to avoid moisture stress. A standard rain gauge on each turntable measures the amount of simulant applied. During treatments, beakers are placed on the turntables to collect samples of simulant for pH and conductivity analysis. Each beaker is placed inside a 30-cm length of PVC pipe on which is centred a large, polyethylene funnel to collect droplets. Replicate samples of each treatment are submitted for analysis of parameters listed under "Simulated Rain Solutions".

Untreated control plants are grown in the greenhouses, but receive no simulants. These are watered with deionized water applied directly to the soil in quantities equivalent to those received by test plants. They also receive the same amount of light as test plants and are placed in a chamber for the same duration as plants which receive treatments. Temperature and humidity are measured in each chamber just before and after a treatment, with an LCD digital thermohygrometer.

Observations and Harvest Procedures

The occurrence of foliar injury is recorded by notes and photographs. Colours are described with Munsell colour charts. Germination, seedling establishment, shoot height, numbers and sizes of leaves, and flower development are also recorded.

In experiments to determine effects on growth or yield, plants are removed from pots with soil intact around the roots. Soil is gently washed from the roots in a plastic tub containing warm water. The washed roots are then dried carefully on paper towels and separated from the rest of the plant. Shoot and root fresh weights are immediately measured to the nearest 0.01 gram. Weighed material is placed in labelled paper bags (one plant per bag) and dried to a constant weight (about 48 h) at 80°C in a forced draft oven. Dried material is reweighed. For very small seedlings, weights may be recorded to the nearest 0.001 gram. Individual specimens may be saved for histological study or herbarium records.

Additional Experimental Facilities

Fumigation Chambers

Four controlled environment chambers are available at Brampton to determine effects on plants of exposure to controlled levels of gaseous pollutants such as sulphur dioxide, ozone, and ethylene, either singly or in combination. Air supplied to these units is filtered to remove air pollutants. Temperature, humidity and day length are controlled. These facilities are used together with the simulated acid rain chambers, to determine the combined effects of acid rain and other pollutants.

Test Garden

An outdoor garden of just under 1 ha has been established at Brampton. Most of this area has a sandy loam soil 30 cm deep over clay. Power is supplied to the site through four outlets and there is a buried water line for irrigation.

Mobile Rain Exclusion Canopy System

A mobile rain exclusion canopy system, located at the Phytotoxicology lab in Brampton, is used to conduct controlled acid rain simulation studies with crops grown in the field under true agricultural conditions. This enables dose-response curves to be derived (yield vs increased rain acidity) for major crops grown in Ontario. Three, mobile greenhouse shelters are used for the exclusion of ambient rainfall, and for the application of simulated acid rain treatments to crops. Soybean and radish crops were investigated during the first year of operation (1984). Future work will examine maple seedlings in pots under the canopies. The potting soil and seedlings for the latter study are obtained from the Leslie Frost Centre near Dorset, Ontario.

Canopies

Three, gothic style, prefabricated, free standing greenhouse structures (dimensions: 19.5 m x 9.1 m x 4.6 m) are constructed of galvanized steel and covered with polyethylene which is anchored in place with aluminum polylocks. Each wheeled canopy sits on a pair of parallel tracks and is moved on and off its respective treatment blocks by means of a cable drive system.

Control Trailer

Control devices, mixing tanks and monitors are housed in a heated control trailer situated behind the canopies. An adjacent enclosure houses the water deionization system, storage tank, treatment reservoir tanks, pumps and electrical panels.

Ambient air concentrations of O_3, SO_2 and NO/NO_2 are measured with monitors situated in the control trailer. Environmental parameters such as temperature, relative humidity, wind velocity and photosynthetically active radiation are measured by sensors located on top of an 8 meter tower attached to the control trailer. Ambient precipitation is measured with a tipping bucket rain gauge situated on the control trailer roof.

Arrangement of Treatment Areas, Control Plots and Nozzle Arrays

Ponty Pool sandy loam was added to a depth of 1 meter in the study area. Tile drains were installed and the area was levelled. Test plants are grown on the three separate test areas (15.2 m x 7.6 m) covered by the rain exclusion canopies. Each block is subdivided into 50 plots (1.5 m x 1.5 m).

Nozzles in each canopy are randomly assigned to two Latin squares and are mounted to the canopy frame at a height of 2.4 m above soil grade. The nozzles are positioned over the centre of each plot when the canopy is in its final position over the treatment block (Figure 2).

Ambient control plots are situated next to the treatment blocks in the area immediately beyond the influence of the canopies. The control plots are on the same soil as the plots within the treatment blocks, but are not covered by canopies and receive only natural rainfall.

Nozzles and Treatment Lines

Specially designed, whirl jet type, stainless steel nozzles emit a square pattern of 1 mm diameter droplets over each treatment plot. Each nozzle is attached to the centre of a plexiglass levelling plate, which can be adjusted for correct distribution over the treatment plot.

Simulated acid rain (SAR) treatments are delivered to the canopies by pumps from treatment reservoir tanks in the trailer. An underground system of PVC pipes leads from the tanks to above ground couplings attached to flexible hoses. The hoses are connected to stainless steel regulators mounted in the south facing corners of each canopy. The flexible hoses enable the treatment lines to remain coupled to the mobile canopies at all times.

A flow regulator is attached in each of five treatment lines to each canpoy. Beyond the regulator, the line is separated into two lines (black polyethylene tubing) each having a PVC needle valve and pressure gauge (0 to 100 kpa) for adjusting flow rates (operating pressure is 30 kpa). One line supplies treatment to five nozzles in the first latin square array, the other line supplies SAR to five nozzles in the second latin square array.

Gaseous Pollutant Reduction System (GPR)

The gaseous pollutant reduction system consists of 9 low pressure, horizontal draw blower fans with 113,500 litre per minute air flow capacity, activated charcoal filters and perforated polyethylene pillows.

Three blower units are situated at the south end of each treatment area (Figure 2). Blower units on two treatment areas are each equipped with pre-filter housings containing 2 Amerframes with 2 M57 media pads and 2 Series 2040 Dri Pak filters. For each blower unit, a side access carbon filter housing is situated in series with the prefilter housing. Each carbon filter housing contains 28 filter trays containing impregnated

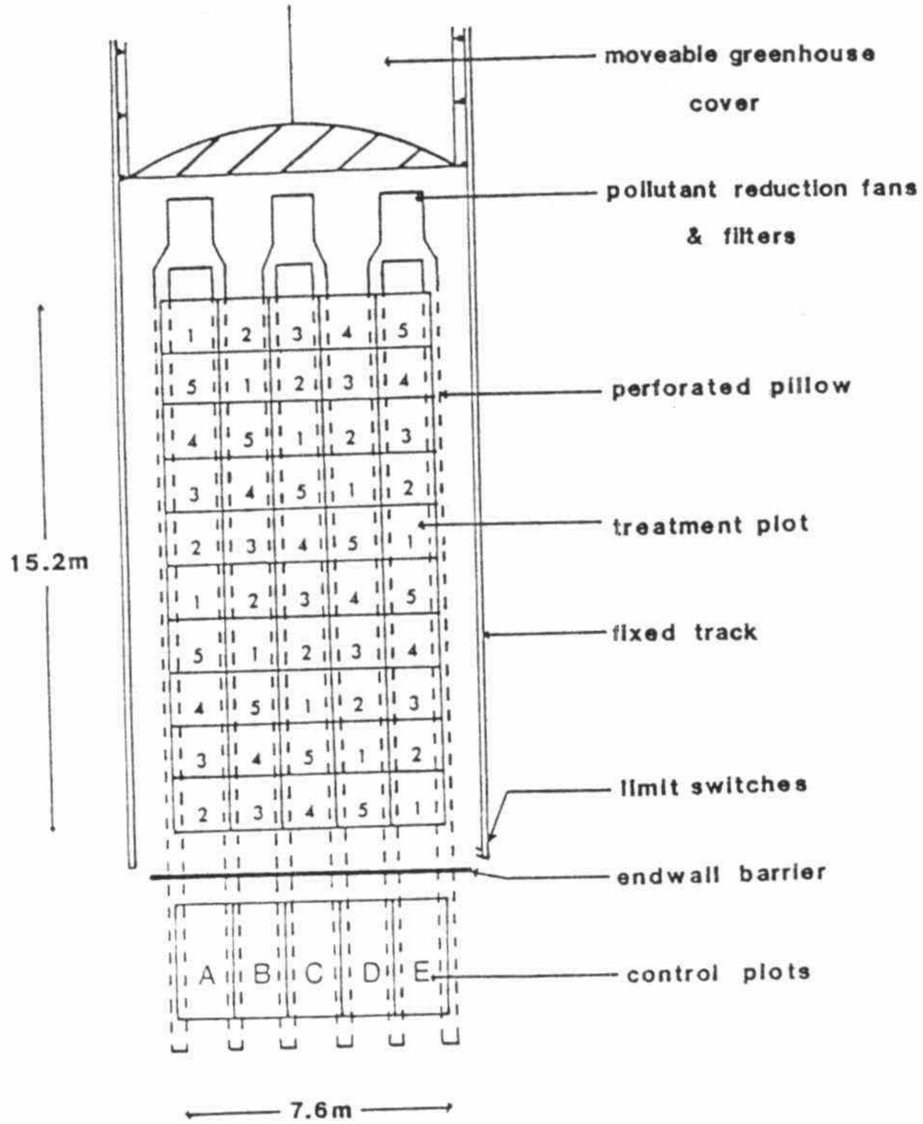


Figure 2. Precipitation Exclusion and Gaseous Pollutant Reduction System (only one system is shown for clarity).

carbon to remove SO_2, O_3, NO_X, H_2S at 90% efficiency. The blower units on the remaining treatment area are not equipped with filters. Consequently, filtered air can be forced over the experimental plots of canopy #1 (west canopy) or canopy #3 (east canopy). Unfiltered air is forced over the experimental plots of canopy #2 (centre canopy) for control purposes.

Air is forced into plots via 30 cm diameter perforated polyethylene tubing or 'pillows'. The pillows extend beyond the treatment blocks covered by the canopies to include ambient control plots which are exposed to natural rainfall (Figure 2). Elevated concentrations of pollutants such as O_3 or SO_2 can be injected into the air exclusion system at controlled rates to determine possible joint effects with simulated acidic rain treatments.

Preparation of Simulated Acid Rain (SAR)

The system has been designed to apply five SAR treatments simultaneously (Figure 3). The SAR treatments are prepared in U.V. stablized plastic mixing tanks located in the control trailer. Treatments are prepared automatically by a system of limit switches, pumps and solenoid valves as required by the computer controller. SAR is prepared in 175 litre batches by dilution of concentrated acid stock solutions into deionized water on a timed basis. Tap water is deionized through a water purification system consisting of a 5 μm prefilter, turbidity filter, tri-bed deionizer columns, carbon filter and 0.3 μm filter. The deionized water (15 megohm resist.) is stored in a 7,500 litre U.V. stablized polyethylene tank. The storage tank is equipped with a U.V. sterilizer for maintaining water quality.

During 1984, treatments of pH 5.6, 4.2, 3.8, 3.4, and 3.0 were used. SAR at the five acid levels is produced by the addition of 240 mℓ of the acid/ion stock solution to 175 litres of deionized water using an electronic metering pump. Separate stock solutions, containing mineral salts and sulphuric/nitric acids based on ambient rainfall chemistry were used (Table 2).

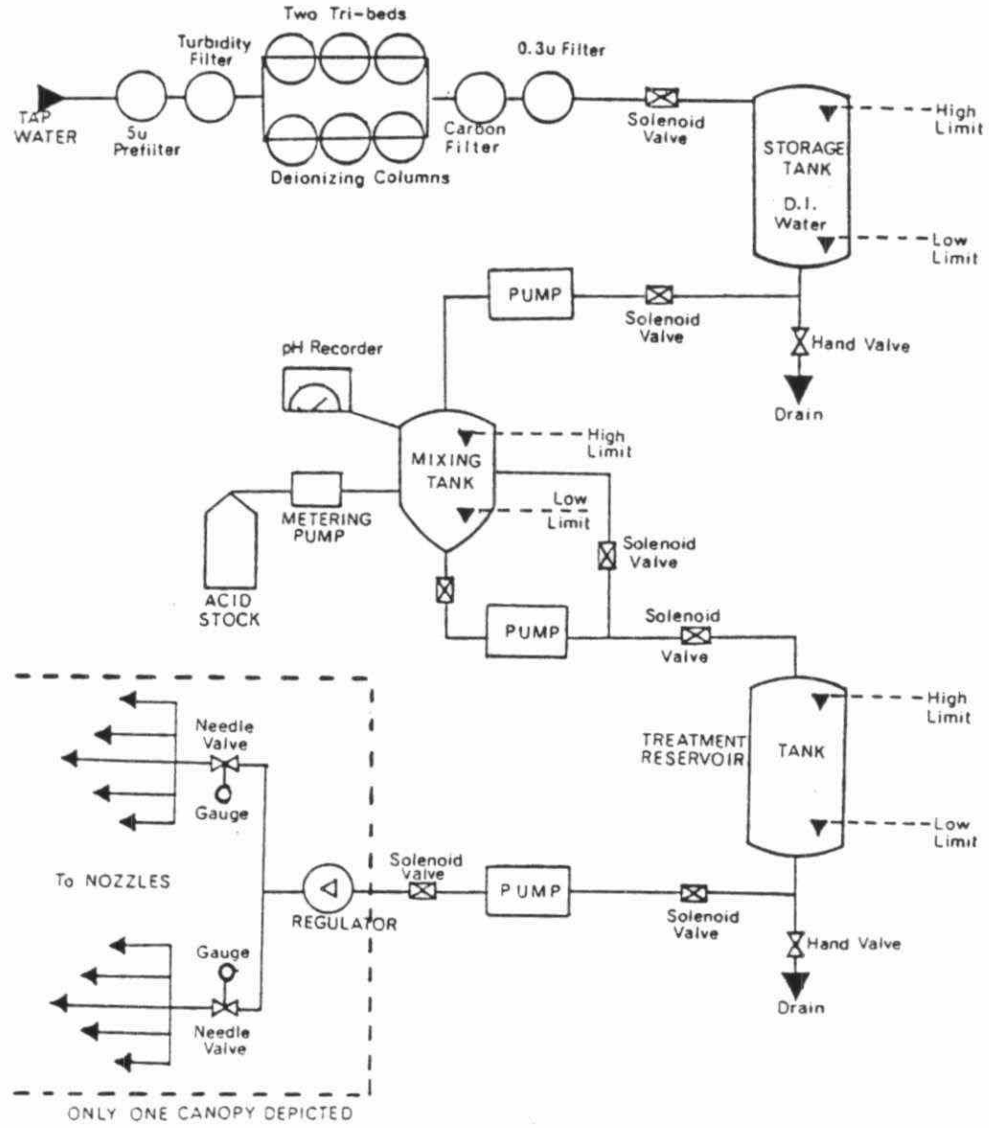


Figure 3. Simulated Acid Rain Application System (only one treatment line is depicted for clarity)

Table 2. Chemical composition of simulated acidic rain (SAR) solutions for Exclusion Canopy Experiments.

pH of SAR	Ion Species Concentration (mg dm^{-3})*			
	H^+	SO_4^{--}	NO_3^-	S/N[@]
5.6	0.003	2.769	1.385	1.99:1
4.2	0.063	4.954	2.476	2.00:1
3.8	0.158	8.245	4.126	1.99:1
3.4	0.398	16.554	8.268	2.00:1
3.0	1.000	37.399	18.684	2.00:1

*All simulated rain solutions contained the following ions: ammonium - 0.744 mg dm^{-3}, calcium - 0.500 mg dm^{-3}, magnesium - 0.097 mg dm^{-3}, sodium - 0.100 mg dm^{-3}, potassium - 0.063 mg dm^{-3}, chloride - 0.023 mg dm^{-3}.

[@] Sulphate to Nitrate Ratio (by Mass).

Each stock solution is specific to each of the five pH treatments. All solutions had a final ratio of 2:1 $SO_4:NO_3$.

In 1985, SAR treatments were used which were equidistant in terms of the hydrogen ion concentration. Acidity levels were expressed as moles or microequivalents of H^+ per litre of SAR. An acid concentration interval of exactly 200 ueq $H^+ \, l^{-1}$ was selected. In addition, the non-acidified SAR treatment was eliminated in favour of an extra SAR level. The acidity levels selected for the 1985 field season are given in Table 3:

Table 3. Acid levels of simulated rain solutions for 1985 field season (crop: soybean).

	H^+ ueq l^{-1}	SAR pH
1.	50	4.301
2.	250	3.602
3.	450	3.347
4.	650	3.187
5.	850	3.071

It is thought that this new SAR chemistry regime will allow the derivation of meaningful dose-response functions and descriptions of the effects of SAR on field grown soybean.

Table 4 describes the concentrations of ions and acids required to acidify 175 litres of de-ionized water. The calculations are based on a dispensing rate of 60 mℓ min^{-1} for a period of 4 min (240 mℓ total) from a 15 litre solution reservoir.

Table 4. Acid (H_2SO_4 + HNO_3) volumes for simulated rain solutions.

A	B		C		D	
pH	mℓ/ℓ SAR		mℓ per 175 ℓ		mℓ per 15 ℓ carboy	
	H_2SO_4	HNO_3	H_2SO_4	HNO_3	H_2SO_4	HNO_3
4.301	1.001E-3	8.849E-4	.1752	.1549	10.85	9.68
3.601	5.002E-3	4.424E-3	.8754	.7742	54.71	48.39
3.347	9.003E-3	7.962E-3	1.5755	1.3934	98.47	87.09
3.187	1.301E-2	1.151E-2	2.2768	2.0143	142.30	125.89
3.071	1.699E-2	1.503E-2	2.9733	2.6303	185.83	164.39

The SAR mixing routine is initiated on a signal from a high limit float switch located in each of five 900 litre treatment reservoir tanks made of U.V. stablized polyethylene. Deionized water is then pumped into the treatment mixing tanks. Thirty seconds after the filling is initiated, the appropriate acid stock solution is added to each tank by a metering pump for exactly 5 minutes. This is sufficient time to give the final pH. Mixing pumps circulate the water in the tanks to insure mixing. The tanks are allowed to mix for an additonal 30 secs and are then dumped into their respective treatment reservoir tanks. The dumping procedure requires an additional 5 minutes to complete.

Computer Controller

All aspects of the field system are controlled automatically by an Apple micro-computer and a Cyborg Isaac data acquisition device. In addition, the computer/controller constantly monitors the precipitation sensor for detection of rain. The total system status is re-checked every 5 seconds for: a) canopy position, b) status of all mixing and treatment application pumps, c) SAR levels of mixing and storage tanks, and d) status of nine blowers (Figure 4).

Temperature, relative humidity, wind velocity, light intensity, and ambient gaseous pollutant measurements are made every

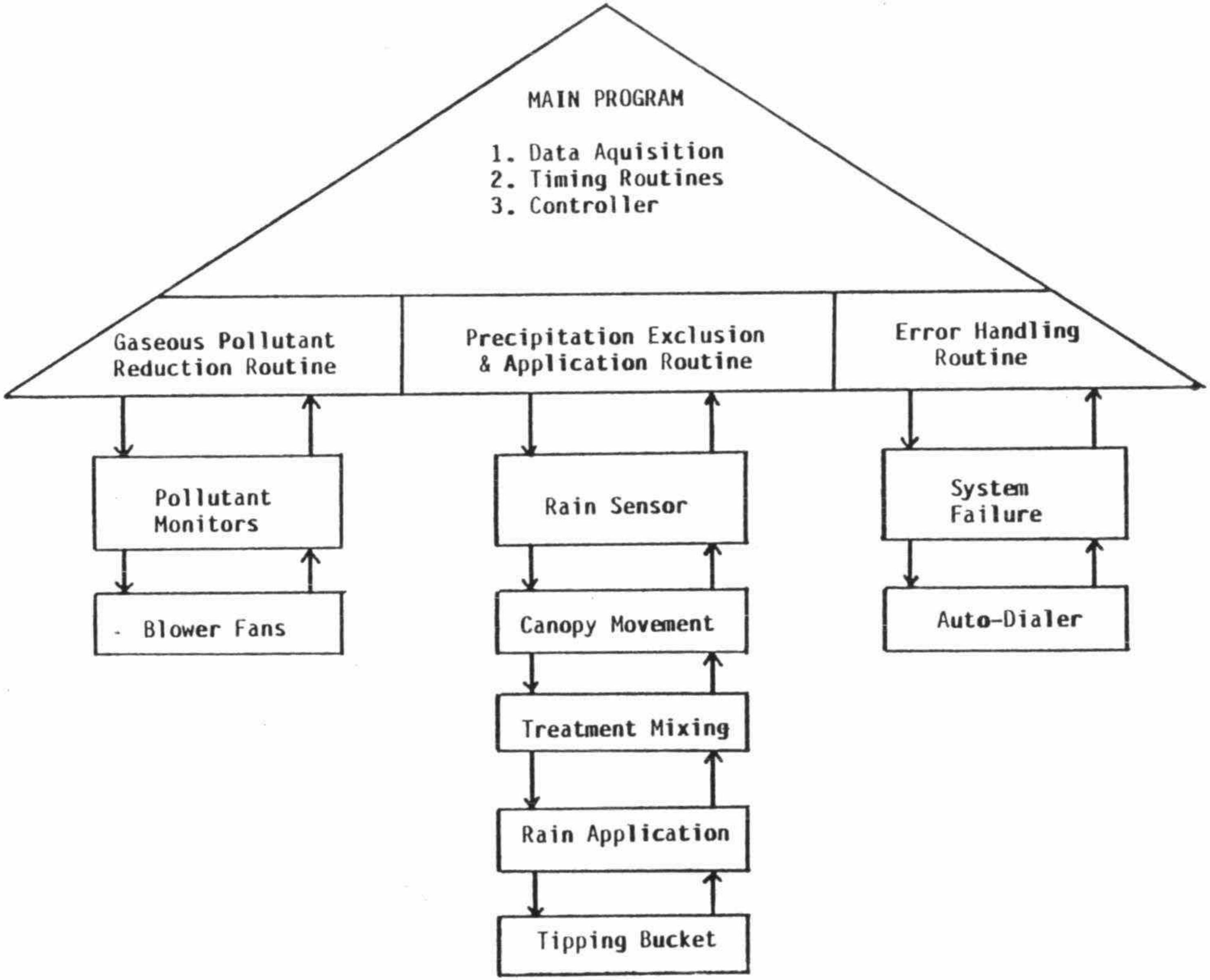


Figure 4. Routines of Computer Controller

10 seconds. Meteorological and air quality data are collected and stored at all times unless a computer failure occurs. Every 30 minutes, previous half hour means of all meteorological and air quality data are stored on floppy disk and then printed for permanent record.

Blowers are activated by the computer controller if a half hour mean value for a given air pollutant exceeds a specified criteria (e.g. 0.05 ppm O_3, 0.15 ppm SO_2 or 0.25 ppm NO/NO_2). Air sampling alternates in 5 minute intervals between ambient air (at trailer) and air sampled from each of the three canopy treatment areas. Blowers are deactivated when precipitation occurs or the half hour mean for all ambient air pollutants fall below the specified threshold level.

When precipitation occurs, the computer immediately signals the microprocessor controlling the canopy drive motors and the canopies move over the treatment areas. (Note: a special routine exists in case of failure). After the canopies are over the test plots, the computer waits until 0.5 mm of ambient rain has fallen before commencing with application of simulated acid rain.

If an ambient rainfall event is low in intensity (hence minimal deposition), the computer controller will apply the identical amount of SAR after the precipitation detector has been dry for a period of 30 minutes. SAR is applied automatically to treatment plots until the amount applied equals the ambient rainfall deposition recorded by the tipping bucket rain gauge. Before applying additional SAR, the computer waits until an additional 0.5 mm of ambient rain has fallen or the ambient rain event has ended.

In addition to the automatic mode, the rain application may be performed manually. If SAR is to be applied when an ambient rain event has not occurred, the computer can be programmed to apply a given quantity of SAR at a given time. SAR is applied in 0.8 mm increments within 10 minute intervals (4.8 mm/hr.) until the total amount requested is applied. The computer maintains the canopies over the treatment plots for a period of 30 minutes after manual SAR application has ended and treatment mixing is completed. Normal program routines such as air quality monitoring and meteorogical data collection are continued during manual mixing and/or SAR applications.

In the event of a failure in any component of the system, SAR application is terminated immediately or will not occur. If operational, movement of canopies on or off treatment plots will continue without interruption. A telephone call is initiated by the computer to inform staff that a failure has occurred in the system.

Field Experiments

A preliminary study was conducted during the summer of 1984 as an initial test of the system. Experiments were carried out to determine the effects of simulated acid rain on the yield of two field grown crops, radish (Raphanus sativus, cv. 'Cherry Belle' and 'Comet') and soybean (Clycine max, cv. 'Bicentennial).

Soybean and radish crops were planted in a latin square design using standard agricultural practices and shielded from ambient rainfall by three moveable greenhouse shelters.

Crops were periodically subjected to simulated acid rain treatments - pH 5.6, 4.6, 3.8, 3.4, 3.0 - in amounts equivalent to the deposition of natural rain events. Control plots were also established which received ambient rainfall only.

Three separate radish crops were grown. 'Cherry Belle' was utilized for the first and third planting; 'Comet' was sown in the second planting.

Radish crops were exposed to 6 (80.2 mm), 8 (113.1 mm), and 6 (81.8 mm) simulated acid rain events during the first, second and third plantings, respectively. The soybean crop was exposed to 23 separate simulated acid rain events for a total of 290.3 mm over the entire growing season.

Soil Preparation and Maintenance

Prior to sowing, the soil in the treatment blocks is plowed, fine tilled and the fertilizer requirements determined. Herbicides and pesticides are applied when necessary. Soil moisture is measured near the surface qualitatively with soil tensiometers, and at depth with a neutron moisture probe. Irrigation must be provided during periods of drought to avoid water stress to the test crops.

Observations of Injury

Foliar injury including frequency, size and location of lesions are noted during the study. The presence and absence of soybean root nodulation is also investigated at a late stage of vegetative development. Tissue samples are collected at regular intervals from test plants on treated and control plots. These are analysed for major nutrient concentrations. Stomatal resistance is also measured.

Harvest Procedures

Radish plants are harvested when mature (approximately 30 days). Every third plant in the centre two rows of each plot within the treatment block is removed until a total of 30 plants from each block have been harvested. Control plots are harvested similarly. Shoots and roots of all plants are gently washed clean and blotted dry with paper towels. Whole plant, fresh weights, and root weights are recorded. Leaf number and area are measured. Root dry weight is determined after drying at 80°C in a forced air oven for at least 48 hours.

Soybeans are harvested when the plants are mature and defoliated. Forty plants are cut at ground level from the centre two rows of each plot within the treatment block. Five replicate samples of forty plants each are removed from each ambient control plot. The number of seeds per pod are recorded on a randomly chosen subsample of 15 plants. Subsample seeds are then combined with the seeds of the remaining 25 plants harvested from each plot. All seeds are oven dried at 40°C for at least 48 hours. The temperature cannot exceed 45°C or the seeds lose their viability. Seed weight is recorded for each plot as a per hundred weight at a standard 13% moisture content, and total dry seed weight in kg/ha is calculated for each plot. Germination tests are performed on samples of harvested seeds to determine percentage seed viability vs. treatment pH.

PROVINCIAL LICHEN AND MOSS MONITORING STUDY

Objectives

Air pollution biomonitoring studies have demonstrated the value of lichens and mosses as bioindicators of deposition contaminants. The lichen and epiphyte monitoring study assesses the value of lichens and mosses as bioindicators of the effects of acidic deposition and related pollutants. Activities include baseline sampling, mapping and modelling of chemical data, providing a lichen and epiphyte inventory of the biogeochemical study areas and examining the feasibility of studying lichen/moss population dynamics.

BASELINE BIOACCUMULATION STUDY

Objective

By sampling and analysing lichen and moss samples throughout the province, a uniform lichen and moss chemistry data base is established. Relationships with precipitation chemistry are examined and future changes can be predicted.

Site Selection and Description

Survey sites are placed as close as possible to APIOS deposition monitoring stations to facilitate comparison of precipitation chemistry with lichen and moss chemistry. Sites are located in forested areas, lacking any visible sign of disturbance, preferably on publicly-owned land.

Baseline sites are located away from local sources of atmospheric contaminants. All sites are at least 100 m from gravel roads, 50 m from paved roads and at least 50 m from the edge of the forest canopy. Approximate site locations are marked on access maps and exact locations are marked on aerial photographs.

Sampling Procedure

At each site, two replicates of each of the following species are collected (where possible): *Cladina mitis*, *Cladina rangiferina*, *Cladina stellaris*, *Evernia mesomorpha*, *Hypogymnia physodes*, *Parmelia sulcata*, and *Pleurozium schreberi*. About 200 g of sample are collected and placed into 1 litre white paper bags. Brown paper bags are not used due to possible contamination from soluble sulphur compounds present in the paper. Plastic gloves are worn during sampling to prevent contamination.

If the moss or lichen must be scraped from bark or rock, a non-metallic implement is used. Samples are cleaned roughly to remove litter and soil. Sample bags are stapled closed, labelled and refrigerated as soon as possible.

Sample Preparation and Submission for Analysis

Plastic gloves are worn when handling samples. Any leaves, bark, twigs, insects, soil or other contaminating substance is removed from the samples. Microbial activity is prevented by drying samples as soon as possible for 24-48 hours in a forced draft oven at 80°C.

Dried tissue is ground with a Wiley mill or under liquid nitrogen with a mortar and pestle until it passes through a 1 mm (40 mesh) sieve. Samples are stored in glass jars with plastic or pulp-lined lids.

Baseline samples are submitted to the Ministry's Toronto laboratory for the measurement of Fe, Mn, P, N, Al, Ca, Cd, Cl, Cr, Cu, K, Mg, Na, Ni, Pb, S, Ti, V and Zn. The LIS test group for these analyses is TEAPS7. The LIS test names are given in Section E. Laboratory Analysis. Standard submission forms are sent with the samples.

Variability Study

A variability study at the Biogeochemical sites examines the effects of growing season on the chemical composition of lichens and mosses.

Data Management and Mapping

Data is stored on a MacIntosh microcomputer. Computer generated contour maps with three dimensional perspective surface are produced to reveal patterns of element bioaccumulation. Contour maps based on Euclidean distance are used to indicate the relationship between precipitation chemistry and bioaccumulator elemental composition.

QUALITATIVE SPECIES INVENTORY

Objective

The objective of this study is to document and assess the condition of lichens and mosses at the biogeochemical study sites, in order to evaluate these sites as cryptogam habitats.

Sampling Procedure

Bulk samples are collected at several locations in each plant community at the biogeochemical sites. If possible, all species of mosses and lichens are sampled from all substrates. A knife is used to scrape or cut lichens and mosses from the substrate. A chisel and hammer are used to sample epilithic species. Samples are placed in white paper bags and labelled with the species, site, data, habitat, substrate, condition assessment and relative abundance. If a sample cannot be removed, close-up photographs are taken.

Sample Identification and Voucher Preparation

For identifications which cannot be made in the field, microscopic or chemical tests are carried out in the laboratory.

Voucher specimens of the species collected are prepared and given to the herbaria of the regional Ministry offices and the National Museum of Canada.

PERMANENT PHOTOMETRIC LICHEN QUADRATS

Objective

Data is collected on the size of epiphytic lichens for comparison with similar measurements in the future. The photographic record also reveals lichen vitality and visual symptoms of injury. The computerized digitizing of this information is important to studying lichen and moss population dynamics.

Site Selection and Description

Permanent lichen quadrats are established at the biogeochemical sites. Twenty permanent 10 x 20 cm quadrats are located on the dominant 2 or 3 tree species at each site. Trees used are free of visible signs of disease or injury and do not lean more than 5°.

All trees are at least 100 m from gravel roads, 50 m from paved roads and 50 m from the edge of the forest canopy. Quadrats are established on the trees over lichens so that they are as nearly as possible centered at 1.3 m above the ground on the north facing side of the tree. Aluminum corner nails mark each quadrat.

Sampling

A photographic sampling technique is used which maintains uniformity of film exposure and magnifications, by employing a shadow box and a ring flash as a light source. A camera is mounted in one end of the box and the quadrat is photographed through a hole in the other end of the box. The camera is locked to a rigid frame to reduce parallax distortion and a microprocessor controlled electronic flash provides exactly the same amount of light for each photograph assuming a uniform grey background.

For the photograph, an elastic white string loop is stretched around the corner nails of the 10 x 20 cm quadrat allowing the scale to be correctly determined on each photograph. A SLR camera, 50 mm lens and Kodachrome ASA 25 film is used. All film is of the same emulsion number.

Lichen Thallus Measurement

A photo image of the quadrat is projected onto a digitizing tablet interfaced to an Apple microcomputer. The area of each lichen thallus is traced on the tablet and the thallus area calculated by the computer. Data is stored on a MacIntosh microcomputer. Lichen cover in these quadrats provides a baseline for future comparison.

DYNAMICS OF LICHEN AND MOSS POPULATIONS

A feasibility study of investigating lichen and moss population dynamics as indicators of vascular plant population changes due to acidic deposition is currently underway. This involves an assessment of the usefulness of techniques for studying moss and lichen population dynamics.

THUNDER BAY DISTRICT LICHEN MONITORING PROGRAM

Objectives

In 1970-71, 75 sites in the Southwestern part of Thunder Bay District were sampled for epiphytes and ground lichens. To determine whether changes in lichen chemistry had occurred over the last decade, 20 of these sites were resampled in 1981 and an additional 20 of the sites were resampled in 1983.

Site Selection and Description

The sites, located on an eight-mile grid, are mostly mixed forest with mesic habitats. White birch, trembling aspen, balsam fir, white spruce and jack pine are common to all sites. Ground and rock lichens are also collected if available.

Sampling Procedure

All sample sites are described. Parmelia sulcata is collected from white birch trees, and Evernia mesomorpha is collected from balsam fir. If suitable open rock areas are available, then Umbilicaria spp. and Cladina mitis are also collected.

Sample Preparation and Submission for Analysis

The samples are hand cleaned and air dried on plastic trays. Samples are ground in a blender to pass through a 250 µm (60 mesh) sieve. Ground samples are stored in glass jars and sent to the Ministry's Toronto laboratory for analysis. The lichens are analysed for the following elements: Al, Ag, B, Ca, Cd, Cl, Cr, Cu, F, Fe, Hg, K, Mg, Mn, Mo, N, Na, Ni, P, Pb, S and Zn. The LIS test names for these elements are listed in Section E. Laboratory Analysis.

B. SOIL STUDIES

BASELINE SAMPLING

Objective

The principal objective of this study is to establish a reliable and uniform data base for soils across the province. The data base will (1) provide current data against which future changes in soil chemistry may be detected, (2) enable development of laboratory experiments to define criteria for the sensitivity of soils to acidification, and (3) provide information required for the sensitivity mapping of Ontario soils.

Site Selection and Description

Baseline sites are located on undisturbed soils. Provincial parks, conservation areas and Crown land offering reasonable access and long-term security are generally best suited for baseline sampling.

To enable the examination of soil chemistry in light of ambient precipitation chemistry, at least one soil site on each important landform is sampled within about a 20 km radius of an APIOS deposition monitoring station. A variety of landforms and soil types are sampled to enable the preparation of soil sensitivity maps. Choice of sampling locations also takes into account variations in acidic input, climate, geology and vegetation.

Site locations are identified by UTM coordinates and a sketch map is prepared showing the sampling location in reference to permanent local features. Sufficient detail is included to permit site relocation. The site is described according to the guidelines in the "Field Manual for Describing Soils" (Institute of Pedology, University of Guelph).

Sampling Procedure

A pit, approximately 1 m^2, is dug to a depth sufficient to permit sampling of the parent material. Soil profiles are described according to criteria adopted by the "System of Soil Classification for Canada" (publication no. 1455, revised 1974, Canada Dept. of Agriculture). The soil pit is photographed and a sketch is drawn of one of the faces sampled to show major soil horizon boundaries and the positions of stones, root masses, and sampling points. Figure 5 is a blank form of the type used to record field data.

Beginning with the deepest horizon, duplicate samples, each weighing approximately 1 kg, are collected with a hand trowel from opposite sides of the soil pit and placed in labelled plastic bags. For special studies, undisturbed soil of known orientation is sampled with Kubiena boxes, or equivalent containers, by carefully pressing the box or container into the wall of the soil pit and removing the sample and container simultaneously. When sampling is concluded, soil horizons are returned to a pit in the reverse order from which they were removed, to achieve the least possible disturbance of the sampling site.

Present plans call for a sampling frequency of approximately once every 5 years for baseline sites in Ontario. To reduce potential effects of seasonal variations on soil properties, repeat samples should be collected at the same time of year as in the original survey, from new pits adjacent to the original ones.

Sample Preparation and Submission for Analysis

To minimize physical and chemical degradation of the soil, samples are spread out on non-metallic trays within a few days of collection and air dried for 48 hours. Dried samples are disaggregated with a porcelain mortar and pestle and screened through a 2 mm (10 mesh) sieve. A subsample of the less than 2 mm fraction is then ground in a motorized grinder with an agate mortar and pestle until the entire subsample passes through a 150 μm (100 mesh ASTM) sieve. The less than 2 mm fraction (at least 150 g) is placed in a glass vial. Both are submitted to the laboratory for analysis. The remainder of the disaggregated sample is stored in a cardboard or glass container of suitable size.

DEPTH
(cm)
HORIZON
DESIGNATION

SITE: ______________

SAMPLE #: ______________

PHOTOGRAPH #: ______________

SAMPLED BY: ______________

SITE #: ______________

LOCATION:

VEGETATION:

DEPTH TO FAINT MOTTLING: ______________ cm

DEPTH TO PROMINENT MOTTLING: ______________ cm

DEPTH TO G LAYER: ______________ cm

DEPTH TO BEDROCK: ______________ cm

HORIZON	COLOUR	TEXTURE	STRUCTURE	HORIZON BOUNDARY	STONINESS	ROOTS

PARENT MATERIAL: ______________

SURFACE EXPRESSION: ______________

SLOPE: ______________

Figure 5. APIOS - Soils Baseline Study Site Report

APIOS-SOILS BASELINE STUDY (CON'T)

DESCRIPTION OF SITE

**SEE: FIELD MANUAL FOR DESCRIBING SOILS (ONTARIO INSTITUTE OF PEDOLOGY)

location: ____________ sample: ____________

sampled by: ____________ site #: ____________

date: ____________

1. slope: simple _____ complex _____

slope class 1 2 3 4 5 6 7 8 9 10

slope percent ________

site position on slope A B C D E F G

2. soil drainage ____________________

3. mottle description ____________________

mottle abundance ____________________

4. pore pattern moisture soil regime: perviousness: rapidly moderately slowly

soil moisture regime: dry _____ fresh _____ moist _____ wet _____

5. forest humus classification ____________________

6. degree of decomposition for organic soils ____________________

7. types and classes of soil structure: type __________

kind __________

class __________

8. area percentage covered ____ ____________________

9. stoniness ____________

10. rock outcrop ____________

The following analyses are performed on baseline samples: pH (distilled water and calcium chloride); exchangeable calcium, magnesium, potassium, and aluminum; permanent charge cation exchange capacity; pyrophosphate and dithionite extractable iron, aluminum and manganese; organic carbon; total carbonates; total nitrogen; plant available phosphorus; soluble sulphur and aluminum; heavy metals (Cu, Ni, Pb, Zn); and texture (particle size). The LIS (Laboratory Information System) test groups for these analyses are TEAPS 6 (Cu, Ni, Pb and Zn) and TEAPS 1 (remaining tests).

SOIL VARIABILITY

Objective

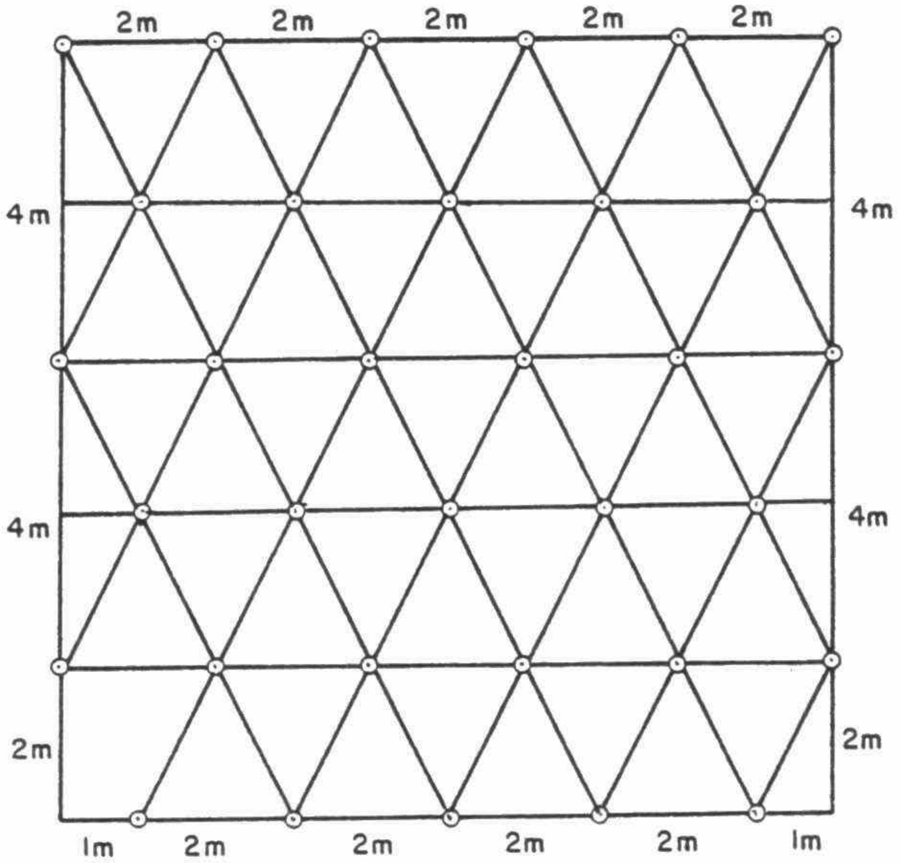
The objective of this study is to document the natural spatial and seasonal variability in some baseline soil parameters at selected study sites.

Site Selection

Within the study area, a representative sampling plot measuring 10 m by 10 m is delineated.

Sampling Procedure

A grid (Figure 6) is superimposed over the sampling plot and 33 sampling points are marked with 25 cm wooden stakes. Using a stainless steel corer, 45 cm long and 30 mm in diameter, at least five soil cores are obtained radially around each stake. Core depth should extend to the C horizon, or to bedrock if no C horizon is present. If the C horizon is below the sampling range of a 45 cm corer, the arm of the corer can be extended to reach the required depth. Each core is separated into its component horizons and the horizons from each of the cores are pooled to form one sample. The sample is then placed in a numbered plastic bag. Because of limitations in resources, the Ministry is sampling only the A and B horizons in its variability study. Study



⊙ Sample site

Figure 6. Sampling Grid for the Soil Variability Study

sites are sampled in the spring, summer and fall over a period of two years.

Sample Preparation and Submission for Analysis

Samples are prepared for analysis in the same manner specified for baseline sampling and the same analyses are also performed. If the sample size is insufficient, top priority is given to the determination of soil pH (water and calcium chloride), exchangeable cations, organic carbon, soluble aluminum and sulphate and texture.

SOIL COLUMN LEACHING EXPERIMENTS

Objective

The objective of these studies is to predict the long-term effects of acidic precipitation on soil under controlled conditions. Laboratory experiments will document changes in soil chemistry and processes resulting from different acid loadings. Study results will be used to develop sensitivity criteria for the preparation of soil sensitivity maps for the province. The data will also be used to predict the number of years for different acidities of precipitation to affect different types of Ontario soils.

Preparation of Soils

Experiments in Toronto use intact columns of A, B and C horizons from three soil types commonly found in southern and central Ontario. Each soil horizon is placed in a buchner funnel (17 cm diameter, 9 cm high) in the field. Horizons are stacked in vertical combinations of A, AB, and ABC horizons. The separation of horizons before treatment permits the identification of horizons altered by acidic solutions.

Similar studies in Thunder Bay use intact profiles (A, B and C horizons combined) from a Brunisolic soil in a watershed study area in northwestern Ontario. Each column is 100 cm long and is contained in PVC tubing, 15 cm in diameter and 120 cm long.

Both the Toronto and Thunder Bay experiments apply simulated acidic solutions at the rate of 500 ml per week. The Toronto application rate represents approximately 1.3 years of average rainfall per year of experiment. In Thunder Bay, the application rate is equivalent to 2 years of rain during 1 year of experiment. The soil leachate from both studies is analysed monthly for major cations, anions, and metals. The sample preparation methods and list of parameters analysed are the same as those for soil leachate in the biogeochemical study.

Equipment

The application apparatus for each soil column in the Toronto study consists of a glass separatory funnel with Tygon tubing attached to its base. The simulated precipitation is drip-fed from this upper reservoir onto the soil surface. The flow is regulated by a series of clamps placed down the length of the tubing. The simulant is distributed over the soil surface by repositioning the end of the tube before each treatment.

The apparatus in the Thunder Bay study differs in its method of simulant application. An inverted polyethylene funnel fits over the top of each column to reduce evaporation from the soil surface. Simulated rain entering the base of the funnel falls on a layer of polyethylene screening (1.5 mm mesh) which lies on a polyethylene disc. The disc is 2 mm thick, 15 cm in diameter, and contains 120 evenly-spaced holes, each 2 mm in diameter. The disc is held in position 10 cm above and the solution which passes through the disc is evenly dispersed over the surface of the soil column.

The time required to feed simulated precipitation into the application apparatus is shortened by attaching each upper reservoir via a series of Y or T connectors to one of three large master reservoirs. These master reservoirs contain different simulated rain solutions. Each upper reservoir is outfitted with a three-way valve at the point of solution entry. The application apparatus is manipulated to produce rainfall rates in the range of 1.27-2.54 cm/h by adjusting the outflow from the upper reservoir using a two-way valve. Rain drops produced by the apparatus are in the range of 2.0 to 3.0 mm in diameter.

Simulated Rain Solutions

A treatment range of pH 2.0 to pH 5.6 has been used in simulation experiments, based on the pH of "normal" rain and the current pH range of rainfall in Ontario. A stock solution is prepared by the Ministry's Toronto laboratory which simulates the average ionic concentration found in bulk precipitation in Ontario. The pH 5.6 solution has the following ionic composition: Na^+ (0.1 mg/ℓ), K^+ (0.06 mg/ℓ), Mg^{++} (0.09 mg/ℓ), Ca^{++} (0.37 mg/ℓ), Fe^{++} (0.01 mg/ℓ), NH_4^+ (0.33 mg/ℓ as N), Cl^- (0.70 mg/ℓ), NO_3^- (0.32 mg/ℓ as N), $SO_4^=$ (0.64 mg/ℓ), and conductivity (9.2 μmhos/cm).

The pH of the solution is then adjusted using sulphuric acid. Samples of simulants and deionized water are routinely submitted to the Ministry's Toronto laboratory for analysis of the same parameters as the leachate.

BUFFERING CURVE STUDY

Objective

The determination of a soil's sensitivity to acidification is critical to the study of the effects of acidic precipitation on terrestrial and aquatic ecosystems. Buffering curve experiments are being done to document changes in soil chemistry resulting from different acid loadings. The relationship of the slopes of soil buffering curves to the physical and chemical properties of soils throughout Ontario will provide a means for establishing sensitivity criteria required for the sensitivity mapping of Ontario soils.

Experimental Procedure

A 10 g aliquot of air-dried soil, sieved to less than 10 mesh, is saturated with 40 ml of reagent, stirred, and equilibrated for at least 1 hour. pH measurements of the soil suspension are made in reagents of increasing acidity (pH 5.6, 4.0, 3.5, 3.0, 2.8, and 2.5) to determine the rate of change of soil pH with inputs of acidity. Nitric, sulphuric, nitric-sulphuric and simulated acidic precipitation reagents are used to assess the response of a soil to different types of acids. A buffering curve is generated by plotting soil pH in each reagent against the concentration of H^+ added to the reagent (Figure 7). To better document changes in soil chemistry during soil acidification, supernatant solutions may be centrifuged, filtered, acidified and analysed for selected base cations and metals.

OPEN-TOP SOIL COLUMN STUDY

Objective

The objective of this inter-regional study is to determine the long-term chemical changes in soil exposed to different climate conditions and different levels of long-range atmospheric pollutants. This information will also supplement the findings of the soil baseline program.

Column Preparation

A total of 72 sections of PVC pipe, each 50 cm long and 15 cm in diameter, were filled with soil. Twenty-four of these columns contained a sandy soil from northwestern Ontario, 24 contained a northeastern Ontario clay soil, and the remaining 24 contained a silty soil from southern Ontario. Each column comprised a well-mixed A and B horizon. A plastic screen was positioned between the A and B horizons and was secured on the bottom of each pipe. Samples of the mixed A and B horizons from each region were sent to the Ministry's Toronto laboratory and analysed for the same parameters listed for baseline soils.

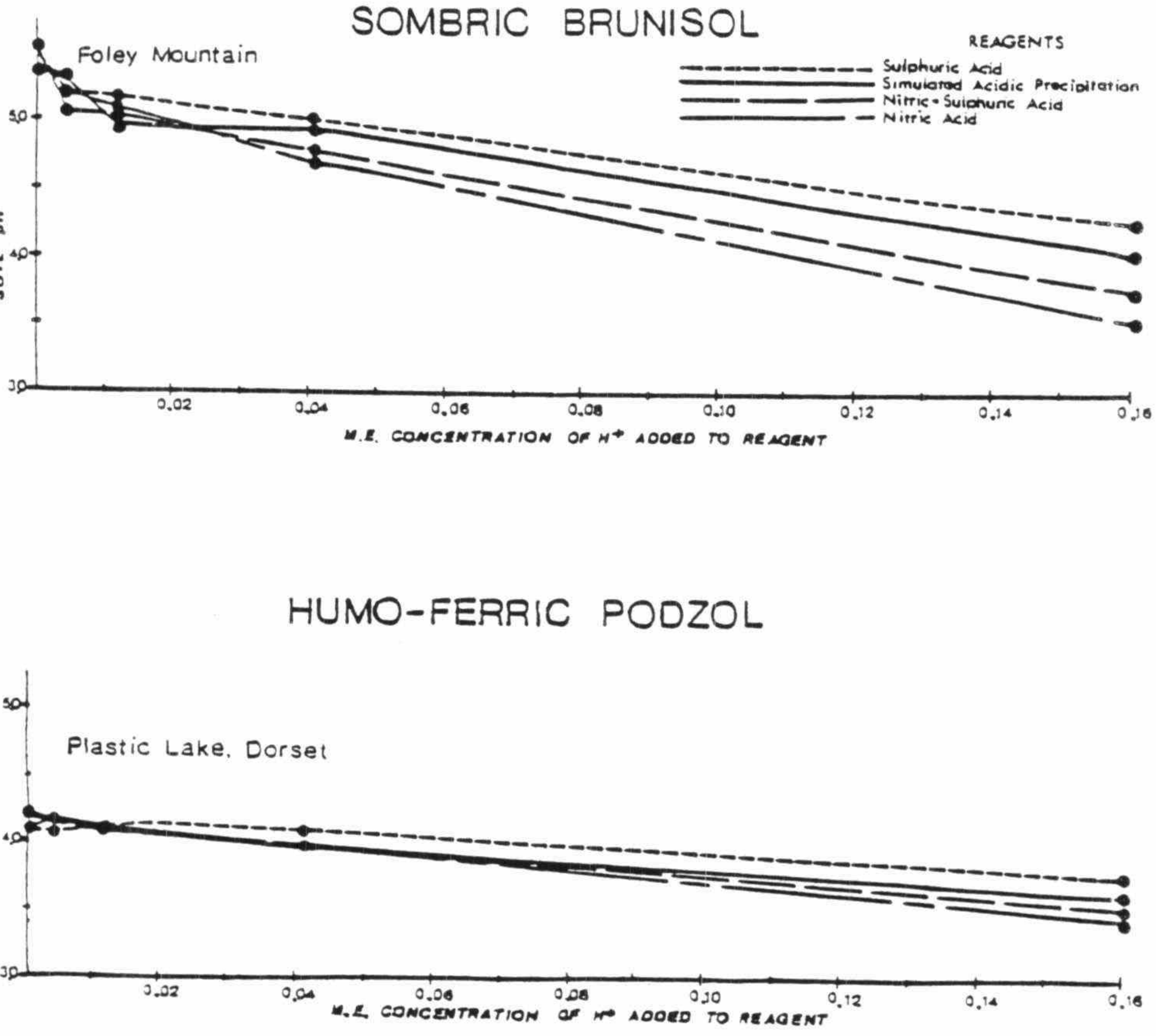


Figure 7. Examples of soil buffering curves of Ah horizons.

Distribution of Columns

The 72 re-constructed soil columns are distributed among three regions of the province (Northeastern, Northwestern and Southern) so that each region has 8 soil columns of a sandy soil from northwestern Ontario, 8 columns of a silty soil from southern Ontario and 8 columns of a clay soil from northeastern Ontario.

The soil columns are placed at field sites selected following the criteria used for A.P.I.O.S. event precipitation collectors. At each site, a trench is excavated to a depth of 60-70 cm. The soil columns are then placed in the trench, B horizon down, with the outer rims of the columns 10 cm apart.

The top of the PVC pipes is 4 cm above the surface of the ground. The trench is carefully filled in and soil is packed down around the pipes. Kentucky Bluegrass is sown on all soil columns and the area kept clear of weeds.

Sample Collection and Analysis

At intervals of 5, 10, 15 and 20 years after the start of the study, duplicate columns of each of the three soil types will be removed from the ground in each region. Soil will be removed from the columns and samples from the A and B horizons will be prepared for chemical analysis following the procedures outlined in baseline soil sampling.

C. BIOGEOCHEMICAL STUDY

INTRODUCTION AND OBJECTIVE

One of the methods currently used to study the environmental effects of acid precipitation is the watershed approach, in which an ecosystem is selected for study based on an area defined by a stream or river drainage basin. The establishment of biogeochemical studies in watersheds so identified is a key component of the investigation of the effects of acid rain on the terrestrial system in Ontario.

The objective of biogeochemical studies is to document element and nutrient distribution and cycling within forested ecosystems subjected to varying degrees of acidic deposition, and to determine the role of forested ecosystems in the process of lake acidification. To achieve the objective, representative components of the ecosystem being investigated must be quantified. From these measurements, simple models of terrestrial ecosystems can be developed, and gains, losses, and cycling of elements within and between the various components in the system can be estimated. By comparing data for areas subjected to different acid loadings, the effects of acidic inputs on the chemistry and biology of the forested ecosystem may be predicted. At each study site, incident precipitation, precipitation throughfall, precipitation stemflow and soil leachate are measured and analysed. Air quality and meteorology is monitored, litter is collected, shallow groundwater is sampled and weir or flume measurements are made. Standing crop biomass, soil mass and litter decomposition rates are determined to assess nutrient storage and accumulation. All these measurements enable the calculation of ionic mass balances. Study sites are located near Dorset, Sudbury and Thunder Bay.

One of the purposes of this manual is to standardize the methodology used at the biogeochemical sites in Ontario. Since the forest types available for study are not uniform across the province, some aspects of the recommended methodology vary among sites, especially with respect to numbers and deployment of monitoring devices. As far as possible, however, field equipment, investigative procedures and sample analysis are standardized throughout all study areas.

SITE SELECTION CRITERIA

The following criteria are considered in selection of biogeochemical study sites, bearing in mind that not every site meet all criteria:

1. Each study area is readily defined within the drainage basin of a headwater stream which flows during a significant part of the non-winter season. The presence of ponds or swamp conditions along the course of the stream monitored could create problems in data interpretation. The recommended size for a study watershed is between 25 and 150 hectares.
2. The two northern Ontario sites are as similar as possible in vegetation composition, particularly with respect to tree species.
3. Soil in each area is "sensitive" to acidification (sandy, low cation exchange capacity).
4. Study sites are subjected to different acidic deposition loading.
5. Study sites, if possible, have similar histories with respect to fire and harvesting. Historical environmental data is useful.
6. All sites are accessible year round, preferably by road, and not influenced by local contaminant sources such as well-travelled unpaved roads. Sites are available on long-term tenure and free of potential vandalism. Access to electric power and a telephone line is desirable.

SITE DESCRIPTION

Detailed descriptions are prepared for all study sites. Site descriptions include a compilation of the following information:

1. Maps: topographical, geological, drainage systems, soil types, etc.
2. Photographs: aerial as well as ground level.
3. Vegetation cover: a plant species inventory list, tree stocking density, ground flora assessment and tree age distribution.
4. Soil type and depth description (Canadian Soil Classification System)
5. Geographic location (UTM coordinates).
6. Climate: annual rainfall, temperature, seasonal wind roses.

To aid in preparing maps delineating the watershed area and its features, a reference grid of 100-metre intervals, coincident with established UTM lines, is useful. The reference grid is used, among other things, to locate sites at which soil depths are measured as these are needed to determine total soil mass. The following method of establishing a reference grid is recommended. A known reference point (A) is established in or near the watershed and is coincident with a UTM (100m) coordinate intersection. A reference line (B) north and south of this point is established to the limits of the watershed and marked at 100-metre intervals. A second reference line (C) running east and west is similarly established. From these lines, the entire watershed is covered with reference points (D) at 100-metre intervals. Figure 8 illustrates this procedure.

MONITORING METHODS

The parameters monitored at biogeochemical study sites have been selected to fit an ecosystem model comprising several

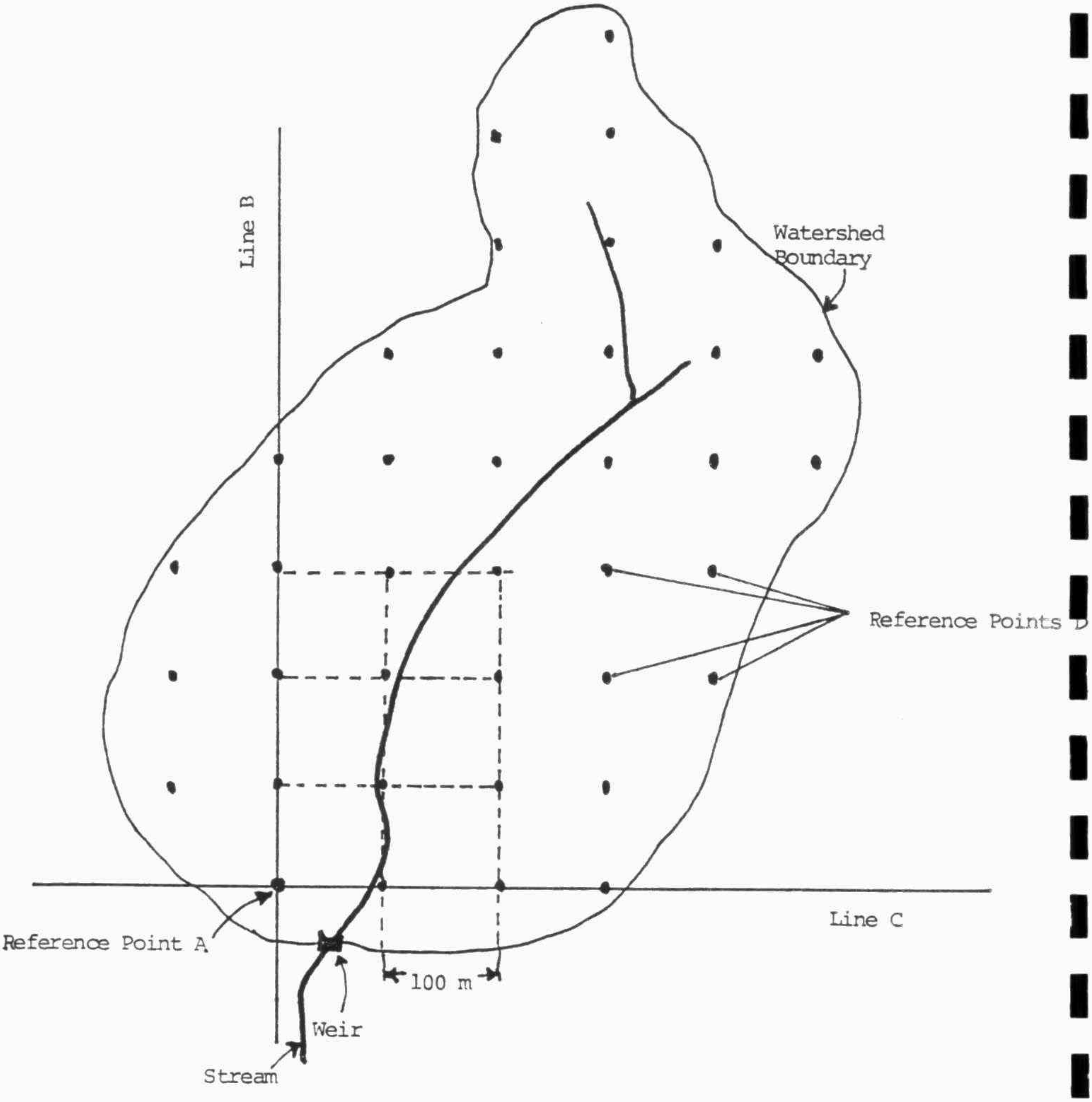


Figure 8. Method for establishing a reference grid on a hypothetical watershed

different elemental cycles. These parameters can be placed into the following four groups according to sampling frequency:

	Frequency	Measurement
1.	Once only	Watershed area, vegetation biomass, soil mass, forest cover, soil zones.
2.	Precipitation event	Incident precipitation, precipitation throughfall, precipitation stemflow, lysimeter leachate.
3.	Fixed interval	Incident precipitation, dry deposition, litterfall, stream water, evaporation rate, groundwater, litter decomposition.
4.	Continuous	Stream flow-rate, SO_2, O_3, NO_x, meteorological measurements (wind speed, wind direction, etc.)

Some parameters are measured with one instrument at a single site in the watershed (eg. air quality and meteorological data), while others require several samplers at different locations. Recommendations regarding sampling locations and equipment installation and operation are described in the following sections.

Watershed Area

The watershed area is determined for use in calculating elemental budgets. The area is established on topographic maps and aerial photographs, followed by ground observations. The watershed boundary is designated by permanent markers on the ground (eg. stakes and/or trees marked with flagging tape or paint) and marked on appropriate maps and aerial photographs. The final area is determined by planimeter or other suitable method.

Vegetation Biomass

Trees

Vegetation biomass is determined to estimate nutrient reserves in the study area so that the response of these reserves to different acid loadings can be assessed. Biomass for trees larger than 5 cm DBH (diameter breast height) is determined from trees representing the range of diameter size-classes in each major species. Dry weights (biomass) are determined for these trees and a regression (biomass equation) of dry weight on DBH establishes mean dry weights for each diameter class. These means are used to estimate dry weights for individual trees in a stand table derived from a forest inventory. The sum of the stand table dry weights gives total stand dry weight per unit area of the watershed. Nutrient contents for whole trees or their components are obtained by multiplying nutrient concentrations with dry weight.

Biomass equations selected from the literature are evaluated for suitability at each study watershed. To do this, at least three trees, representing the smallest, median and largest DBH classes encountered, are destructively sampled for each major species in each watershed. For each tree felled, fresh weight is determined in the field for cones, living branches, and dead branches. Subsamples of each of these components are obtained for subsequent dry weight measurement and chemical analysis. The tree stem is cut into 10 sections of equal length. Fresh weight is determined for each section, and a disc, 2-4 cm thick, is cut from the base of each section. In the laboratory, fresh weights are determined for subsamples of cones, dead branches, live branches with attached foliage, and stem discs. These samples are dried at 80°C to constant weight. For live branches with foliage, dry weights are determined separately for foliage and branch material. Once the moisture content and weight ratio of foliage to branch are known, the total dry weight of foliage and branches may be determined for each tree. Dry weights are also measured separately for stemwood and stem bark. Data for moisture content and for bark-to-wood ratio enable total dry weight to be estimated for tree stemwood, including bark. Discs representing the first, fourth, and eighth sections of the stem are analysed for nutrient content.

The weights determined for each of the sample trees per species are compared with weights calculated from biomass equations in the literature. If actual and estimated weights differ by no more than 10 percent for the whole tree, or 25 percent for any of its components, then the existing equations are adopted.

If suitable biomass equations are not available from the literature, they are developed for each study watershed. To avoid disturbance in the watershed, sample trees are felled in a nearby similar area. About 20 trees per species are required. Sampling is carried out as described above. Regression equations are developed to relate dry weights of whole trees, or their components, to DBH.

Similarly, if no suitable equations are available for below-ground (root) biomass, roots of 10 to 15 trees per species, representing the full range of diameters, are manually or mechanically excavated. Excess soil and stones are washed from the roots with water. Cleaned root systems are cut into three categories: stumps, roots > 2 cm diameter, and roots < 2 cm diameter. Total fresh weight of each of these categories is determined in the field. Suitable subsamples of each category are obtained for moisture content determination and chemical analysis in the laboratory, following procedures outlined for above-ground biomass. Root biomass equations are then developed by relating dry weights to stem DBH. To reduce the laborious process of root excavation if several major species are involved, root biomass may be estimated by interpolation. In this case, only three trees per species, representing the smallest, median, and largest diameter class, are sampled. Root biomass of individual trees in the stand table are estimated by interpolation from these values, then summed to give total root biomass per unit area.

Samples are submitted for chemical analysis in accordance with the procedures outlined under Submission of Samples for Chemical Analysis (p. 64).

Small Trees, Shrubs and Herbaceous Flora

If present in sufficient quantity, the biomass of small trees, shrubs, and herbaceous flora is determined. Circular plots, each 10 m^2 (radius 1.78 m), are established for small trees and large shrubs (between 1 and 5 cm DBH) in different stand types, while 1 m^2 quadrats are suitable for smaller vegetation. Stems in circular plots are recorded by species and by 1 cm DBH classes. A representative sample of each species is harvested for analysis. For small specimens, the whole plant is obtained. Large specimens are divided into foliage, stem, and root. From quadrat plots, all living vegetation (including roots) is harvested and divided, as appropriate, into (a) shrubs and tree seedlings, (b) herbaceous plants and (c) grasses. For all sample material from both plot types, fresh weight is determined in the field. Dry weight is obtained after sample material is dried at 80°C to constant weight in the laboratory. After chemical analysis (same as tree biomass samples), the total nutrient reserve is calculated for each vegetative type in the watershed.

Soil Characterization

Surveys are conducted in the study watersheds to classify and map soil types, and to obtain data on soil nutrient reserves. These reserves must be known if the potential impact of acidification is to be assessed.

At intervals no greater than 100 m along grid lines established in each watershed, soil cores are obtained to C horizon or bedrock. Additional cores are collected between grid intersections if a significant change in vegetation or topography occurs. Soil texture, type and depth of horizon, depth to bedrock (in shallow soils), drainage, colour, the presence of stones, mottles, and carbonates are recorded for each sample core. The most important of these features (soil texture, horizon type, and horizon depth) are plotted on a watershed map. Areas with similar horizon types and depths are then identified. In areas where horizon type and depth are similar, three or four soil pit sampling sites are selected. Each soil pit represents equal areas within its horizon type and depth. More soil pits may be required if soil horizons and depths are highly variable.

Soil pits are 1 m^2 in size. Within the designated pit area, the L, F, and H horizons are sampled from a 25 cm^2 area. Using a 25 cm^2 wooden frame as a guide, the LFH horizon is cut out with a knife, removed and retained for analysis. This sample includes mosses and lichens which may be growing in the litter, but not higher plants. The total thickness of the LFH horizon on each of the four sides of the wooden frame is recorded. The soil pit is then excavated, described, and sampled as outlined for baseline soil sampling (Section B). In addition to samples for chemical analysis, cores are obtained with a standard bulk density sampler from the middle of each horizon exposed in the pit.

To determine the weight of different soil horizons, a soil pit of 0.5 m^2 is excavated near the larger pit. Each horizon, after it is excavated, is weighed in the field, then sieved through 75 mm and 20 mm screens to segregate stones. The weights of the 20-75 mm stones and >75 mm stones are recorded separately for each horizon. Any significant amount of soil adhering to the stone fractions is removed and weighed. If this is done in the laboratory, samples of about 20 kg for 20-75 mm stones, and 100 kg for stones >75 mm, are required. A sample (approximately 5 kg) of soil in the <20 mm fraction is also obtained and weighed.

In the laboratory, bulk density samples are dried to constant weight at 105°C (48 hours). The different size fractions of samples from soil horizons in the 0.5 m^2 pit are also dried and weighed. The <20 mm size fraction of soil is sieved through a 2 mm screen to remove stones in the 2-20 mm size range. The volumes of particles between 2 mm and 20 mm, between 20 mm and 75 mm, and larger than 75 mm are calculated by assuming a density of 2.65 g/cc.

The volume of each soil horizon is calculated by multiplying horizon depth with horizon surface area. Volume is then multiplied by weight per unit volume (bulk density) to yield total weight. The weights of the stone fractions are subtracted from this figure. Soils are prepared and analysed for the standard LIS test groups TEAPS 1 plus TEAPS 6 (p. 65). Data are used to classify the soils in the watershed under the Canadian System of Soil Classification (Can. Dept. Agric. Publ. 1646, 1978). Total and available nutrient levels in soils will be determined in the future. When these data are available, nutrient storage reserves for each horizon in a soil profile can be estimated. From this, soil nutrient reserves for the whole watershed can be calculated.

Incident Precipitation (Event)

Precipitation events are measured with a standard rain gauge and collected using an "Aerochem Metric" precipitation collector and precipitation throughfall collector. These samplers are installed according to siting criteria adopted for the APIOS deposition network. If a clearing of suitable area cannot be found in or near the watershed to be investigated, then the samplers are mounted on a tower above the tree canopy.

Samples are collected after each distinguishable rain period. This period may be less than an hour or may last for several days. Once the rain period has ended, samples are collected within 24 hours. Precipitation volume and pH are then measured and recorded and the sample submitted for analysis (p. 64). Samples which cannot be submitted immediately to the laboratory are refrigerated until shipment.

Precipitation Throughfall

Throughfall is measured with polyethylene funnels, each having a 650 cm^2 (approximate) collection area. The funnels have a vertical rim (finished to a knife-edge) to reduce splash effects, and are supported so that the rim is approximately 1 metre above ground level. The stem of the funnel is inserted into a plastic dustfall jar lid in which a hole of suitable size has been drilled. The inside of the jar is lined with a polyethylene bag to collect the sample. The funnel stem opening is covered with a nylon screen of 100 mesh, and "Parafilm" is wrapped around the outside of the stem to seal the space between the stem and the hole in the dustfall jar lid.

Collectors are placed under representative tree species and tree size classes. To obtain samples representative of the given canopy, more than one collector may be required under large trees. In such cases, collectors are placed at different distances from the tree stem. Samples are collected as specified for event samples of incident precipitation. Samples are pooled for each tree monitored, volume and pH are determined, and the samples are then submitted for analysis (p. 64). At the biogeochemical sites in the two northern regions, tree species occur in groves. To reduce the number of chemical analyses required, samples are bulked as a proportion of their contribution to the total grove volume to produce one sample. Every 10 to 14 days, immediately after a precipitation event, funnels are removed for cleaning in the laboratory with "Acationox" (Canlab) detergent, followed by a triple rinse with distilled deionized water or equivalent. Funnels removed from the field for laboratory cleaning are replaced immediately with a spare set, to ensure collection of all dry deposition between events.

Throughfall under shrub canopies is collected with a plastic trough placed on the ground. The trough is fabricated from black, ABS 50 mm diameter piping or equivalent, split lengthwise. The trough may be a single length or have three or four side branches. A collection area of at least 0.25 m^2 is recommended. The trough is inclined slightly to conduct collected throughfall to a dustfall jar placed below ground level. The jar is lined with a collection bag of the same type specified for throughfall samplers. Figure 9 illustrates one possible configuration.

Precipitation Stemflow

Stemflow is measured with collection collars attached to designated trees in the watershed. Sufficient collectors are used to adequately represent the different tree species and size classes occurring in the area.

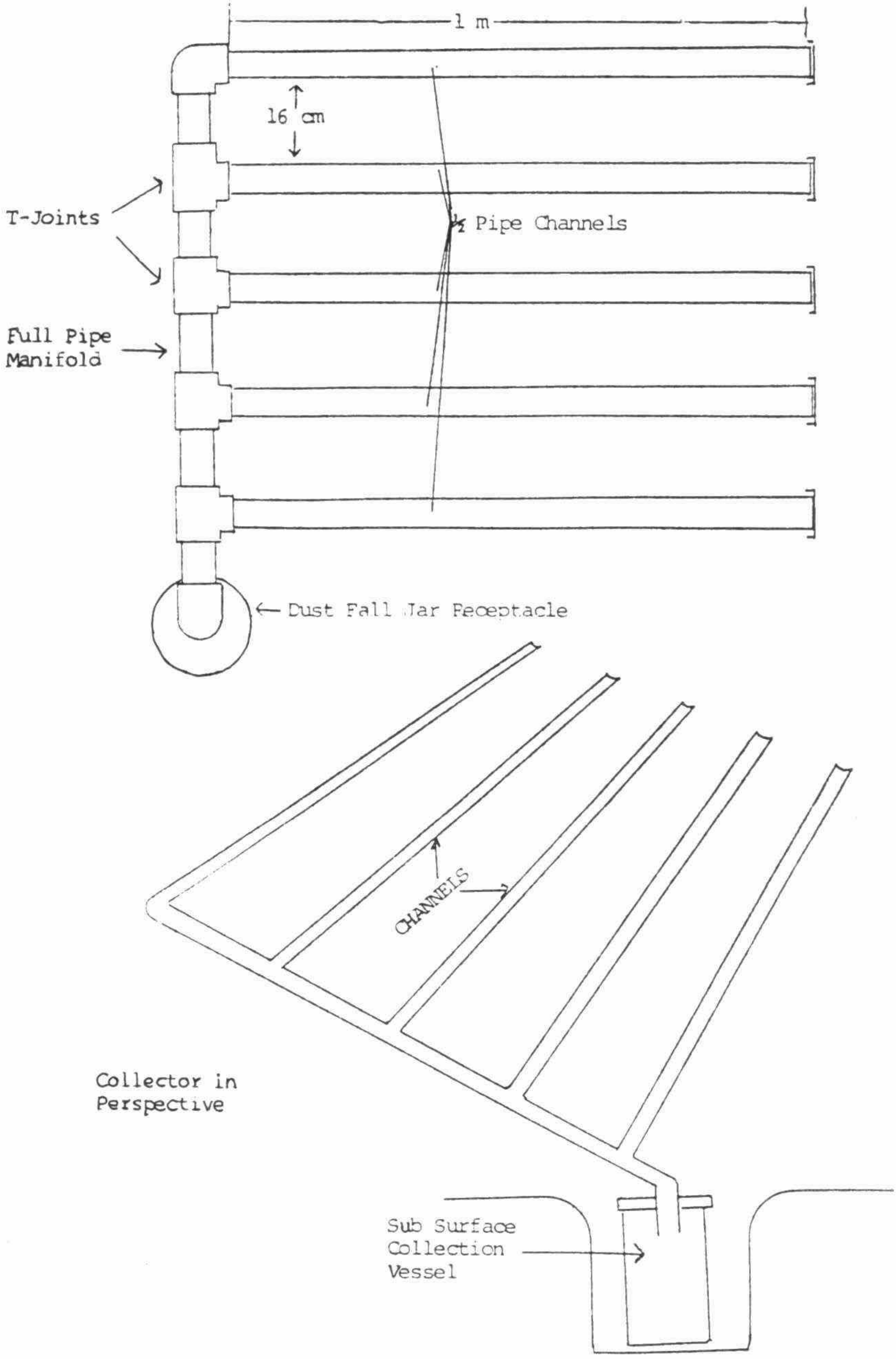


Figure 9. Shrub Throughfall Collector

A collection device consists of split "Tygon" tubing (Formulation R3603, ½ to 1-inch diameter) wound 1½ times around the tree stem between 1 and 2 metres above the ground, and which drains into a collection vessel. To achieve good contact with a tree stem, some of the outer bark is removed. The tubing is fastened to sample trees with stainless steel staples. After installation, a second cut is made in the tubing approximately 0.5 to 1 cm wide. The tubing is sealed to the tree stem with acrylic caulking. Stemflow from the tubing is collected in large plastic garbage cans lined with plastic garbage bags of suitable size.

The timing of sample collection is the same as that for incident event precipitation and precipitation throughfall. Collected samples, if large, may be weighed in the garbage can. A 475 ml sample is removed for analysis (p. 64) and the remaining stemflow is poured on the ground around the bole of the tree sampled. In the northern regions, stemflow samples are bulked as described for throughfall.

Tension Lysimeter Leachate

Alundum/ceramic plate tension lysimeters (15 cm diameter) are installed in the vicinity of representative tree species and size classes. Trees other than those sampled for throughfall and stemflow are selected for lysimeter studies and should be appropriately marked. A soil pit is dug under designated trees in the same manner specified for soil baseline studies. During this procedure, soil removed from different horizons is piled separately on plastic garbage bags or plastic sheets, to keep the surrounding area clean. Suitably cleaned lysimeter discs are installed at the bottom of the A and B horizons and approximately 15 cm below the upper boundary of the C horizon. (Dorset tension lysimeters were installed in the middle of the A and B horizons, but future lysimeters will be installed at the bottom). Cavities to insert the discs in the wall of the soil pit are made with minimum disturbance of the soil. Discs are installed horizontally, porous side up. A paste of soil and distilled water is applied to the upper surface of each disc to create a suction interface with the soil above. Discs in the different horizons are offset from one another so that no disc lies directly above another. After discs are installed, sample tubing is drawn to the surface, and the soil is carefully returned to the pit in the reverse order from which it was removed. Tubing from each lysimeter disc is labelled, capped, and, if necessary, covered with protective material (eg. PVC piping) to prevent rodent damage.

Samples of lysimeter leachate are removed from the soil with a constant vacuum pump which applies a vacuum of approximately 0.1 bar across the lysimeter. Samples, whose volumes are individually recorded, are combined for a period of up to one week, and then submitted for analysis (p. 64).

Zero Tension Lysimeter Leachate

Gravitational soil-water is sampled by a modified Jordan through-type zero tension lysimeter (acrylic tubing cut in half, 25 cm long; 15.5 cm wide outer diameter; capped with nylon mesh). Samples are taken below each of the F, A and B horizons at stations within the upper, middle and lower slope at a monitoring site in a study watershed. The lysimeters are installed in duplicate per horizon by digging a soil pit/trench (about 2.5 m long x 1.5 m wide across the slope) and tunneling into the bottom of the A and B horizons in the upper slope face of the soil pit/trench. Soil removed from different horizons is separately piled on plastic sheets to minimize horizon mixing and disturbance to the surrounding area. The lysimeter is wedged against the top of the tunnel and soil is tamped around the lysimeter. Below the F layer, the thick organic mat is cut and peeled back. A groove is dug, the lysimeter inserted and the organic mat rolled back. If the organic layer is thin, the LF layer is scraped away, the lysimeter inserted in a groove and the scraped LF layer replaced. Lysimeters are not installed directly above one another.

Leachate samples move by gravity through Tygon tubing from the lysimeter's drainage pipe to the collection carboy. If the soil is deep, the carboy is placed below the lysimeter. A retaining wall and folding cover (plywood) houses the carboys. If the soil is shallow, the carboy is located farther down the slope allowing the leachate to flow into the carboy. The Tygon tubing is buried to prevent rodent damage by putting the excavated soil back into the trench. Before the tubing is buried, it is inserted into a plastic drainage pipe to prevent it from collapsing under the weight of overburden soil. Leachate samples are submitted for analysis (p. 64).

Incident Precipitation (Cumulative)

Where electric power is available, "Sangamo" precipitation samplers are employed in the collection of cumulative samples at 28-day intervals, as specified for the APIOS deposition network. Sampling procedures and laboratory tests requested are those adopted by the APIOS network. Air Resources Branch staff is consulted for advice concerning sampling equipment.

Low Volume Air Sampler

At study sites offering a power supply, monthly measurements of selected atmospheric nitrogen and sulphur compounds are made with a low-volume air sampler. The type of sampler and its operation is the same as that used in the APIOS network. The operation of a low-volume sampler is omitted if a similar device is already present at a nearby APIOS deposition site.

Litterfall

Litterfall collectors consist of wooden frames supporting 1 mm mesh polyethylene screening. Each collection surface is either 0.5 or 1 m^2 in area and the wooden sides extend approximately 15 cm above the screening. The collector is positioned horizontally with the screen 15 cm above the ground. Stainless steel staples attach the screen to the frame.

Litterfall collectors are positioned in one of two ways: (a) at each of the stemflow and throughfall sample stations in each watershed. The collectors are located one-half of the distance between the trunk of the tree and the drip line of the tree. The selection of trees under which the collectors are placed should reflect the size distribution in the tree population; (b) randomly throughout the watershed. Method (b) is preferred in watersheds where heterogeneous mixtures of trees, rather than pure stands or groves of trees, are found.

Samples are collected at least six times per year: 1) after snow melt, 2) after tree flowering, 3) prior to autumn colour change, 4) midway through autumn leaf fall, 5) end of leaf fall and 6) prior to snow accumulation.

Sample material is separated into several categories. All categories should be present midway through and at the end of autumn leaf fall but may not be present at other times. The categories are 1) foliage of major species (separated by species), 2) female reproductive organs of major species (separated by species), 3) other foliage (minor tree species, shrubs and herbs), 4) other female reproductive organs, 5) twigs, branches and bark and 6) miscellaneous material (bud scales, conifer seeds, basal sheaths, male reproductive organs, etc.)

Where there is only one dominant tree species contributing to the litterfall collection, there may be as few as four categories of material: foliage of major tree species; other foliage; twigs, branches and bark; other miscellaneous material.

After separation, sample material is placed into small paper bags and dried to a constant weight at 80°C in a forced draft oven.

Individual dry weights for each category are recorded for each litterfall collector. Common categories from each collector are pooled and ground in a Wiley mill to <1 mm, in preparation for chemical analysis. If there is sufficient material in any category for replicate chemical analysis, then that material is split before it is ground. Samples are then submitted to the laboratory for analysis (p. 65).

Leaf-Litter Decomposition

Leaf-litter decomposition is estimated by dry weight loss and nutrient loss in foliage exposed to natural decomposition in litter bags.

In the autumn, freshly fallen leaves of major tree species in each of the study watersheds are collected separately. To obtain sample material from short-needle coniferous species, which shed their foliage throughout the year, nylon mesh litter traps may be placed under piles of freshly cut branches. This process yields sufficient sample within about a month. Foliage-bearing branches from short-needle conifers may also be force-dried to obtain sample material.

Litter bags are 27 cm by 25 cm and are made from nylon mesh. The empty bags are weighed before foliage is inserted. Approximately 15 g of foliage is added to each bag, along with an identifying plastic tag. Each bag is closed with stainless steel pins fastened at the upper left and upper right corners, and at the upper middle section of the bag. If stainless steel pins are unavailable, nylon thread of suitable strength may be substituted. The filled bags are dried at 60° C to a constant weight. Five litter-bags are randomly selected from each species and submitted for initial chemical analysis (p. 65).

In the field, a sufficient number of bags are set out to permit collections of five replicates per species, three times a year, for three years. The field experiments commence in the autumn. Small plots of bags are established on the ground under an overstory of the same species represented by the bags. In the Dorset area watersheds, more than one species is represented in each litter-bag plot. In the northern Ontario watersheds, each litter-bag plot contains only the one species found in the pure stand or grove in which the bags are placed. Plots are also set out to assess litter decomposition on slopes facing different directions.

Before the bags are placed on the ground, the natural, newly-deposited litter is removed. The bags are laid out in a square or rectangular plot, with each bag about 25 cm from its neighbour. For plots containing more than one foliage species, the species are randomly mixed within the plot. The bags are secured to the ground with a netting of 5 cm mesh nylon, staked at the corners, which covers the whole plot. The plot is then covered with fresh litter to achieve an effect similar to its natural surrounding. During the experiment, measurements of soil temperature and moisture are taken. A maximum and minimum soil thermometer is inserted in the leaf litter layer, and a soil moisture tensiometer is placed at a depth as near as possible to the litter layer. Readings from both instruments are taken after every rainfall or at least weekly.

Three collections of litter-bags (5 bags per species) are obtained during the year: early spring, mid-summer and late autumn. Litter is removed from the bags, sampled and dried at 60 °C to constant weight. Samples are submitted for chemical analysis (p. 65).

Stream Water

Measurements are made of stream flow rates and water chemistry. Flow rates are monitored continuously at a stream gauging station comprising a weir or flume, stilling well, and water level recorder. More than one weir may be necessary for each biogeochemical site. If only one weir is used, it is located near the point where the stream leaves the study watershed. Water Resources Branch staff or specialist Regional staff are consulted for advice regarding weir installation and operation. Several technical publications are also available for guidance.

Samples for chemical analysis are collected once a week at a point approximately 10 metres upstream of the weir. During periods of high flows, more frequent sampling is required to more accurately determine losses of ions and nutrients from the study area. Sampling and analytical procedures follow, as closely as possible, those used by Water Resources Branch at the Dorset study sites.

Evaporation Rate

An estimate of water lost from the study area by evaporation and transpiration is required to establish a water budget. Evapotranspiration can be estimated from temperature data with the Thornthwaite system. Evaporation is determined with standard Class A evaporating pans. Water Resources Branch staff should be consulted for advice.

Groundwater

Water table levels and groundwater quality are monitored. Because soil features (especially depth to bedrock) may vary significantly between sites, Water Resources Branch or Regional staff are consulted before proceeding with equipment installation and measurements. Piezometers and standpipes provide data which assist in determining a water budget for the watershed. Groundwater level measurements and groundwater samples for chemical analysis are taken at least once a month.

Air Quality Monitoring

Continuous measurements of SO_2, O_3, and NO_x in ambient air are obtained with monitors approved by the Ministry and housed in a suitable shelter at a site in or near the study area. The contractor operates the monitors under the direction of Ministry staff, but repairs and audit calibrations are carried out directly by the Ministry. If existing monitors are already in operation in the vicinity, monitors are not required at the biogeochemical site.

Meteorological Measurements

In addition to precipitation measurement and analysis, continuous monitoring of wind speed and wind direction is recommended. Every effort is made to comply with accepted criteria for siting wind monitoring equipment. Significant interference from the forest tree canopy and local geographical features is minimized.

Other meteorological data which may benefit the biogeochemical study include air temperature and relative humidity, net radiation, soil temperature, and measurements of precipitation periods and precipitation intensity.

WINTER PROGRAM

Most of the field work at the biogeochemical study areas with the exception of snow sampling is suspended during winter months.

Snow Sampling

Snow sampling is done to provide data on precipitation flux during winter months. Incident and throughfall snow are collected at the same sites where incident and throughfall rain are sampled. The collection efficiency of incident snowfall is determined with a Nipher-shielded snow gauge.

The sample collectors are large, plastic refuse containers lined with polyethylene bags. A polyethylene ring is inserted inside the lined container, just below the rim, to obtain a fixed, reproducible collection surface area.

Samples are collected weekly. If insufficient snow has been collected in a week to yield at least 500 ml of meltwater, the collectors are left exposed for a second week. If snowfall is occurring on the scheduled collection day, sample pick-up is deferred for a day. Each sample is weighed on a spring scale. A new liner is inserted in the collector and the ring replaced. Samples are melted at normal indoor temperatures. If melt-water volume is less than 3 or 4 litres, and if no leakage has occurred, a more accurate sample volume is determined in the laboratory.

During early spring and late autumn, precipitation may alternate between rain and snow. During such periods, both rain and snow collectors should be active. Field staff decide which type of sample to collect, based on the predominant type of precipitation. Any sample in the non-used collector is discarded.

Snow meltwater samples are submitted to the laboratory for analysis (p. 64).

Other Activities

Also during the winter, the sites are visited frequently (perhaps weekly) to undertake the following work:

(a) Performance of routine checks on all continuous air quality and meteorological monitoring equipment (including electric generators, if used).

(b) Performance of routine checks on stream flow recording equipment.

(c) Regular measurements of snow pack depth throughout the watershed area. Snow core samples are collected, following normal Ministry sampling procedure, just prior to and during the spring snow melt period.

Data Records

To ensure completeness of records of the biogeochemical study, the following suggestions are offered as a guide:

(a) Photographs of equipment installations throughout the study area.

(b) Maintenance of a visit log book to record details of study activities, including unusual observations. Separate log books will also be maintained for use with all continuous monitoring equipment.

(c) Appropriate sample shipment records, chemical analysis request forms (LIS), and records of precipitation sampling.

(d) All data forms for the biogeochemical study are standardized throughout the province.

(e) Keeping detailed documentation of all field procedures, eg. sample collection, quality assurance procedures, etc.

SUBMISSION OF SAMPLES FOR CHEMICAL ANALYSIS

All sample locations are identified with a 7-digit code compatible with the location codes assigned under the Ministry's laboratory information system (LIS).

Samples are collected and submitted in accordance with procedures outlined in the Ministry's "Outline of Analytical Methods" (1981, Laboratory Services and Research Branch), the "Field Procedures Manual" (1983, Air Resources Branch) and the APIOS "Terrestrial Effects Procedures Manual" (1983), unless specifically instructed otherwise. Each sample is identified with its own number, from a series allocated by the Phytotoxicology Section, Air Resources Branch, and is submitted on LIS "Request for Analysis" forms.

All aqueous samples to be analyzed for trace metals are filtered through a 0.45 micron "Millipore" filter, preserved with sufficient ultra pure concentrated HNO_3 to obtain a solution of 1% HNO_3 in the sample (approximately 20 drops per 60 ml sample, or 10 drops per 30 ml sample), and submitted in 100-ml plastic vials supplied by the Ministry's Toronto Laboratory stores. For remaining tests, filtered samples are submitted unpreserved in a 475 ml polystyrene jar with a plastic lid. All aqueous samples are refrigerated until shipped. Batches of incident and throughfall precipitation including snow melt-water samples may be submitted under one submission number, but stemflow, leachate and streamwater sample batches are each submitted separately. The following tests are performed on all aqueous samples except soil leachate: total soluble Al, Fe, Mn and Zn, volume, pH, Gran acidity, reactive SO_4, Cl, NO_3, Ca, Mg, Na, K, NH_3-N and PO_4. The corresponding LIS test groups are SXITC for soluble trace metals and TEWQ for the remaining parameters. The LIS test names are given in Section E. Laboratory Analysis.

Aqueous samples of leachate from lysimeters are analysed for pH, Ca, Mg, Na, K, SO_4, NO_3, Cl, Al, Fe, Mn and Zn. The corresponding test groups are SXITC for Al, Fe, Mn and Zn and TESX for the remaining parameters. The LIS test names are given in Section E. Laboratory Analysis.

All soil samples are air dried, disaggregated with a mortar and pestle and passed through a 2 mm sieve. A subsample of this fraction is placed in a labelled glass jar. A subsample of the less than 2 mm fraction is ground in a motorized grinder with an agate mortar and pestle until the whole subsample passes through a 150 µm sieve. This fraction is placed in a glass vial with the same number as its corresponding less than 2 mm fraction. Both samples are submitted for analysis.

All soil samples are initially analysed for pH-water pH-$CaCl_2$, exchangeable calcium, magnesium, potassium and aluminum; permanent charge cation exchange capacity; dithionite extractable iron and aluminum; sodium pyrophosphate extractable iron, and aluminum; organic carbon, soluble aluminum, water soluble sulphate, total nitrogen, total carbonates, acid extractable copper, nickel, lead and zinc, and particle-size. The LIS test groups for these analyses are TEAPS 6 for the acid extractable trace metals and TEAPS 1 for the remaining tests. LIS test names can be found in Section E. Laboratory Analysis. Later in the study, as information on soils from biogeochemical sites is accumulated, the number of analytical tests may be reduced or the tests requested may be changed.

Vegetation samples are dried in a forced draft oven at 80°C to a constant weight. Dried samples are ground in a stainless steel Wiley mill until the whole sample passes through a 1 mm screen. The sample is stored in a glass jar with a plastic or pulp-lined lid. All samples are analysed for total P, K, N, Cl, S, Ca, Mg, Na, Al, Mn, Fe, Cu, Zn, Mo, Ni, Pb and Cd. The LIS test group for these elements is TEAPS 5. Corresponding LIS test names can be found in Section E. Laboratory Analysis.

D. FOREST PRODUCTIVITY AND DECLINE STUDY

MUSKOKA MAPLE DECLINE STUDY

Objective

The objective of this study is to determine the role that acidic precipitation is playing in the decline of sugar maple trees in Ontario.

Site Selection and Description

A total of eleven sites are examined. In response to complaints from members of the Ontario Maple Syrup Producers Association, seven maple woodlots were chosen for study in the Muskoka-Parry Sound Districts of Southern Ontario. Two of the seven woodlots had not been tapped for maple syrup production. These seven sites provide a gradient of dieback conditions as well as representing different management practices, and histories of disease and insect damage. One unmanaged site which was not affected by forest tent catapiller but which was similar in soil type and acidic deposition, is located in Algonquin Park. A "control" site, in a lower acidic deposition area but also with the thin podzolic soils, is located near Thunder Bay, Ontario in the Northwestern Region. Two sites on calcareous soil but also with reports of maple decline are located near Peterborough, Ontario.

Study Methodology

At each site, a permanent observation plot (20 m x 20 m) is established. All trees greater than 10 cm are tagged and numbered. Crown condition and tree health of all species within the plot is assessed based on a rating system (Figure 10). Height and diameter at breast height (DBH) measurements are also made. Adjacent to the permanent observation plot, six trees (2 healthy, 2 moderately affected and 2 severely affected) are chosen for sampling purposes. Height, DBH and crown conditions are assessed

Figure 10. Plot Assessment Form for the Muskoka Maple Decline Study.

SITE NO.: ____________ Weather: ____________

Surveyors (initials): ____________________________

1. frost cracks
2. conks
3. open wounds
4. insect infest.
5. mites
6. cankers
7. s. root rot
8. tap holes

Tree No.	Species Code	Diam (cm)	Height (m)	Angle (deg)	Dist (m)	Percent Dead Branches	Percent Slight Chlorosis	Percent Strong Chlorosis	Crown Class Rating	Additional Observations							
										1	2	3	4	5	6	7	8

NOTES:

CROWN CLOSURE (%): ____________

in the same manner as the permanent plot trees. Foliage and twigs are sampled with pruning poles. Surface litter and mineral soil (0-20 cm) are sampled from around each of the six sampling trees using a soil probe. Increment cores are taken at DBH from the northeast and southwest sides of all 6 trees for growth ring width and elemental analysis.

Two of the six trees (one healthy, one severely affected) are cut down and cross sectional discs are obtained at 2 metre intervals along the length of the tree stem for growth and volumetric calculations. Foliage is collected from the top and the bottom of the Crown. After careful excavation, roots less than 1 cm are collected from those trees which were felled, and separated into 2 subsamples. The very fine roots (less than 2 mm) are collected for elemental analysis and the remaining roots (2 mm to 1 cm) are collected for starch analysis and elemental analysis. All roots are frozen immediately after collection until processed. Replicate soil samples are taken from around the sampled roots.

A soil pit is dug at each site adjacent to the permanent observation plot in an area representative of the study site. Soil is sampled and described according to criteria adopted by the Canadian System of Soil Classification (1978). The sampling procedure is described in Section B. Soil Studies. Analysis of these samples will provide an indication of the sensitivity of the soil to acidification. Selected parameters from the soil collected around the sampled trees is compared to those from the soil pit to provide an indication of local soil variability within each study site.

Acidic deposition rates, management practices, history of disease and insect defoliation, site disturbance, tree age and site quality are investigated at each of the 11 sites to determine the role acidic deposition is playing in the decline of sugar maple trees in Ontario.

Sample Preparation and Submission for Analysis

Foliage and twigs are dried at 80°C in a forced draft oven and ground to less than 1 mm in a Wiley mill. Root samples are placed in a warm ultrasonic bath for 30 minutes to remove soil attached to the roots. The roots are then dried and ground as described above. All vegetation samples are submitted for the measurement of Fe, Cu, Zn, Mo, Ni, Pb, Cd, P, K, N, Cl, S, Ca, Mg, Na, Al, and Mn (LIS test group TEAPS 5) and also for B, Co, Cr, F, and V. The LIS test names for these elements are given in Section E. Laboratory Analysis. Root samples are also submitted for starch analysis. For this analysis, a subsample of the ground sample is extracted three times in 80% ethanol and dried overnight. Twenty milligrams is then weighed into a test tube. The samples are submitted for analysis in the tubes (see Section E. Laboratory Analysis).

Soil samples are air dried disaggregated with a mortar and pestle and passed through a 2 mm (ASTM 10 mesh) sieve. A subsample of the less than 2 mm fraction is ground in an automated grinder with an agate mortar and pestle to pass through a 150 µm (ASTM 100 mesh) sieve. Samples are submitted for measurement of the same parameters as the foliage and twigs as well as the tests done on the baseline soil samples (see Section B. Soils Studies).

Increment core samples are mounted on a board and submitted for elemental analysis by PIXE (Proton-Induced X-ray Emission). A duplicate core is retained for age measurement.

HARDWOOD DECLINE SURVEY

Objectives

The hardwood decline survey was established in 1985 to determine the extent and severity of decline in selected hardwood species throughout the Great Lakes - St. Lawrence and Deciduous Forest Regions of Ontario. The study provides baseline information on the current condition of hardwood trees in Ontario at a series of permanent observation plots located throughout the province. Repeated annual observations may demonstrate future trends in forest decline and the health of hardwood species in Ontario may be extrapolated from observations made at the plots. Initially, sugar maple and its associated tree species will be examined. This study is intended as an inventory of decline and will not necessarily establish any cause-effect relationship with acidic precipitation.

Site Selection and Description

A total survey area of 200,000 sq. k is divided into 100 km X 100 km UTM grid blocks with 2 to 4 sites located in each grid. An appropriate number of plots are allocated to each forest section based on the area of the forest section relative to the total study area. Field plots are located after consultation with Ministry of Natural Resources staff in the appropriate areas of the province. All sites meet the following criteria:

(a) a semi-mature to mature sugar maple stand, representative of an area over 10 ha in size.

(b) a relatively undisturbed area (no cutting, grazing, etc. within the last 20 years).

(c) good access.

(d) sites are located on lands in public or private domain subject to permission of access and study activity, and reasonable assurance of tenure by present landowners or manager.

(e) at least 10 km from known point sources of pollution.

(f) where possible, the stand contains at least 40 trees of a preferred associated species such as beech, birch, aspen or ash within each plot.

(g) the plot area is at least 50 m from any edge effects.

In 1985, 52 plots were located and 41 plots were installed. In 1986, further sites will be established to a total of 110 permanent plots.

Sampling Procedure

Plots are laid out on site using standard forestry procedures. A plot centre is fixed and located from a fixed reference point using standard surveying techniques. From this centre, the location of all nearby trees greater than 10 cm in diameter are plotted using polar coordinates, in a radius moving outward until 100 trees are tallied, including at least 50 sugar maple and approximately 20 of the two tree associates present. Each tree is identified with a permanent tag and referenced to the plot centre on a stand table projection map. The plot centre itself is located by the placement of a 4 inch X 4 inch tagged post in the ground.

Each marked tree in the plot is individually assessed and the field conditions at the site are described according to the forms shown in the Appendix.

Data Management

A decline Index based on the percentage of dead branches in the crown, percentage of undersized leaves and evidence of chlorosis is calculated for each of the 100 trees on each plot. A mean Site Decline Index and within plot variability are determined for each site. Regional patterns are identified, if apparent, and their relation to zones of air pollution determined.

A data file with site descriptions and tree conditions is maintained. Portable remote data terminals equipped with modems are used to record the data on a daily basis in the field. Data is transferred to an IBM personal computer.

DENDROCHRONOLOGY STUDY

Objectives

At the same time the Hardwood Decline survey was initiated, a dendrochronology study was established with the following objectives: (a) to develop regional growth chronologies for sugar maple in three forest sections within the Great Lakes -

St. Lawrence Forest Region using stem analysis; (b) to determine the variability in sugar maple growth patterns on a plot-by-plot basis through DBH increment coring in addition to the stem analysis; (c) to determine if a decline in sugar maple has occurred or is occurring and to determine whether relationships are apparent between sugar maple growth chronologies and environmental conditions, including airborne pollutant levels; and (d) to determine whether or not significant differences occur between the growth chronologies in different forest sections and between different site (soil) types. The three forest sections of interest in the Great Lakes - St. Lawrence Forest Region are: #2 Upper St. Lawrence, #4b Algonquin-Pontiac, #10 Algoma.

Site Selection and Description

As airborne pollutant level is the variable of interest in this study, care is taken to minimize variation of other environmental parameters. Plots are located in close proximity to each other within each forest section in order to minimize within forest section climatic variability. Within each forest section plots are located on three different site types, each type having soils of different buffering capacity. This allows growth variation due to differing soil types within each forest section to be measured.

Sites are chosen to meet the criteria outlined in the Hardwood Decline Study. Each 10 m X 10 m plot contains approximately 10 trees, assuming a mean 3 m by 3 m spacing. If this is not obtained, plot size is increased. The plots are equally divided into sub-plots for increment coring only and sub-plots which require destructive sampling (stem analysis). All plots are assessed using the forms shown in the Appendix.

Sampling Procedure

Increment cores are taken from all trees on a north and south axis at DBH (i.e. two radii are obtained) and stored in labelled plastic straws for TRIM analysis (tree ring increment measurements).

Two trees (dominant and codominant) in each plot are selected and destructively sampled for stem analysis using the TRIM method. Trees of similar form, preferably having a single dominant main upper branch, are chosen throughout the study. The north side of each tree is marked with a permanent marker and the trees are felled between ground level and 15 cm above the ground. Discs are cut from the trees at 40 cm intervals and labelled sequentially from the base of the tree. The DBH disc is specifically labelled, as it is used for comparison with the increment cores. Discs are placed in labelled kraft envelopes, and all discs for each tree are placed in separate burlap sacks. Discs are frozen until TRIM analysis can be performed. As TRIM analysis was developed primarily for softwoods, a methodology for sugar maple will be developed concurrently with this study.

A minimum of 10 soil cores are collected from within the dendrochronology plot. Each core is 0 to 30 cm in depth (minus the organic layer). The 10 cores are bulked in a clear polyethylene bag identified by a unique sample number and stored in a "pic-nic" type cooler until delivered to the Phytotoxicology Section, Air Resources Branch, Ministry of the Environment. Samples are air dried and prepared for analysis according to procedures outlined in Section B. Soil Studies. Samples are analysed for pH, % sand, % silt, % clay, exchangeable Ca, Mg, K, Al, CEC, soluble SO_4 and Al, total N, organic carbon and carbonate.

Data Management

Portable remote data terminals equipped with modems are used. Data is transferred to an IBM personal computer data base management system. The TRIM software is used to generate growth curves and growth chronologies for different site types, forest sections and airborne pollutant levels. Growth patterns will be related to site characteristics, forest section and historical pollution and environmental data.

ANALYSIS OF TREE GROWTH STUDY

Objective

The effect of acidic precipitation on tree growth has never been properly documented in Southern Ontario, partially due to an inadequate historical data base. A historical data base, however, does exist for part of the Muskoka-Haliburton region, and may be useful in the examination of tree growth over the past two decades. This study, therefore, investigates tree growth and stand dynamics over the last twenty years and its possible relationship with acid precipitation in the Muskoka-Haliburton region of Ontario.

Site Selection and Description

The Faculty of Forestry, University of Toronto, maintains eight permanent sample "woodlots", in an area near Dorset, Ontario. The sample "woodlots", each approximately 43 acres in size, consist of six compartments of approximately 7.2 acres each. Three compartments in each woodlot have had all trees 2.5 inches BDH and larger marked and measured in 1962. In 1972, all trees were remeasured. Four of the 24 compartments were measured in 1983. Woodlots 2 and 5 have been chosen as the focus for this study and were remeasured in 1984 and 1985. Woodlot 2 is predominantly hardwood with a minor softwood component. Woodlot 5 is primarily coniferous and lies within the boundaries of the Plastic Lake subwatershed associated with the Biogeochemistry study. Studies on these woodlots, therefore, complement this study.

Sampling Procedure

In 1962, trees in woodlots 2 and 5 were marked and measured as described above. Marked trees were measured in 1972 and 1983. Work in this study includes: subdividing woodlots 2 and 5 systematically into one-tenth acre units for remeasurement, remeasurement including the recording of growth, mortality and missing trees; renewal of tree identification markers, and stem analysis.

Stem analysis is done on a range of DBH species to relate current tree growth and stand information to earlier stand development. In 1984, approximately 10 trees of each of five different species (white pine, hemlock, beech, sugar maple and red maple) were destructively sampled in areas adjacent to the woodlots and sectioned for tree growth analysis. Discs were removed at 1/10 height intervals and at DBH, providing 11 discs per tree. In 1985, an additional 12 trees from each major species were sampled from both woodlots. Growth measurements are also calculated from subtended angle measurements between each tree and its surrounding trees. Detailed information on the project can be obtained from W. Gizyn, Air Resources Branch.

E. LABORATORY ANALYSIS

INTRODUCTION

The sample workload for the Terrestrial Effects Acid Precipitation in Ontario Study (APIOS) is split between the Water Quality Section (WQS) and the Inorganic Trace Contaminants Section (ITC) at the Ministry's Laboratory Services Branch, Resources Road, Rexdale.

The ITC Section analyses all APIOS soil and vegetation samples as well as aqueous samples requiring trace metals analysis, and Water Quality Section analyses water samples for the remaining parameters. A brief outline of these methods is given in the following pages. More detailed descriptions of the methods can be obtained from: A. Neary, Dorset Research Laboratory, Ministry of the Environment (soils analysis); Liz Pastorek, ITC Section, Laboratory Services Branch (vegetation analysis); P. Vijan, ITC Section, Laboratory Services Branch (waters samples for trace metals); F. Tomassini, Water Quality Section, Laboratory Services Branch (water samples for remaining parameters).

WATER SAMPLES

Total Iron, Manganese, Aluminum and Zinc

(LIS test names: FEUT, MNUT, ALUT, ZNUT)

Incident precipitation, and field filtered throughfall, stemflow and lysimeter leachates are preserved with 1% HNO_3. Incident precipitation, stream and snow samples and soil leachates are preconcentrated and analysed for Fe, Al, Mn and Zn by flame atomic absorption spectrophotometry. Throughfall and stemflow samples are analysed without preconcentration by sequential plasma spectrophotometry.

Chloride, Sulphate, Nitrate, Phosphate, Ammonia, pH, Volume, Gran Acidity, Calcium, Magnesium, Potassium, Sodium.

(LIS test names: CLUT, SSO4UR, NNO3UR, NNHTFR, pH, VOLUME, ACDG, CAUT, MGUT, KKUT, NAUT)

Aqueous samples are analysed for the above parameters by the Water Quality Section of the Laboratory Services Branch. A detailed description of these methods is given in the "Handbook of Analytical Methods for Environmental Samples". (Ontario Ministry of the Environment, 1983). Briefly, pH is done electrometrically; volume is done in the field, Ca, Mg, Na and K are analysed by atomic absorption spectrophotometry, Gran acidity is done by titration with NaOH to the inflection point; SO_4 is measured by ion chromatography; and Cl is done colorimetrically on stemflows and soil leachates and by ion chromatography on throughfalls and precipitation samples. Ammonia-nitrogen and NO_3-nitrogen are done colorimetrically on all aqueous samples.

SOIL SAMPLES

pH

(LIS test names: PHEW, PHECA)

pH is defined as the negative logarithm of the hydrogen ion concentration and is a measurement of hydrogen ion activity. The expression of pH in logarithmic units means that a change in pH of one unit represents a tenfold change in H^+ concentration. The pH is one of the most informative soil characteristics and may indicate possible nutrient toxicity or deficiency.

A 10 g sample of air-dried less than 2 mm soil is weighed into a tube and 20 ml of distilled water are added (PHEW). Samples are shaken on a reciprocating shaker for 20 minutes and left to equilibrate for 30 minutes. The pH is then determined electrometrically. The test is repeated using 0.01 M $CaCl_2$ instead of distilled water (PHECA). The $CaCl_2$ reduces interference due to liquid junction potential, flocculates the soil to reduce interference from suspended material, standardizes the electrolyte concentration and brings it closer to that of the soil solution, decreases the electric double layer so that the solution pH more closely represents soil pH, and has a pH closer to rainwater in equilibration with atmospheric CO_2. Because of the advantages of the $CaCl_2$ method, pH measured in distilled water is primarily used for comparison with historical data.

References:

McKeague, J. A. 1978. Manual on soil sampling and methods of analysis. 2nd ed. Canada Soil Survey Committee on Methods of Analysis, Ottawa; 66-68.

Hesse, P. R. 1971. Soil chemical analysis. John Murray Pub. Ltd. London; 19-36.

Particle Size

(LIS test names: SAND, SILT, CLAY)

Particle size is one of the most stable soil characteristics and controls porosity, permeability, water holding capacity, leaching potential, cation exchange capacity, weathering behaviour, and geotechnical aspects of the soil. It is a useful parameter when determining the sensitivity of soil to acid rain.

The measurement of particle size is restricted to the inorganic phase and is carried out by pipette analysis. A 10 g sample of air-dried less than 2 mm soil, is pre-treated for carbonate and organic matter removal using sodium acetate (pH 5) and hydrogen peroxide, respectively. The remaining sample is dispersed with Na-hexametaphosphate-Na-carbonate and the sand fraction (<53 μm) is removed by wet sieving and is then dried and weighed. Measurement of the silt and clay fraction is based on Stoke's Law of settling velocity. Particles of a specific average size are collected by pipetting aliquots from predetermined depths at predetermined times. Samples are dried and weighed. Percentage sand (>53 μm), silt (53-2 μm) and clay (<2 μm) are calculated.

References:

Day, P. R. 1965. Particle fractionation and particle size analysis. Methods of soil analysis. Part 1. C. A. Black et. al. (eds.) Agronomy Monograph No. 9, Madison, Wisconsin: 545-567.

Green, A. J. 1978. Particle size analysis. Manual on soil sampling and methods of analysis. J. A. McKeague (ed.). Canada Soil Survey Committee on Methods of Analysis, Ottawa; 4-13.

Total Carbonates

(LIS test name: CCO3UR)

Carbonates occur naturally in soils, usually in the form of calcite and dolomite (calcium and magnesium carbonates), although many other forms may exist. Carbonates are not usually present in significant amounts in soils whose pH is less than 7 and are non-existent in soils whose pH is less than 5. Because carbonate neutralizes acid, its measurement is important in acid rain studies.

This method is based on the measurement of CO_2 evolved by the reaction of carbonate with 2N HCl in a closed system under constant temperature and pressure. The CO_2 evolved is swept by purified air through a KI scrubber into the cathode compartment of a coulometer. The evolved CO_2 is absorbed by the cathode solution and measured by automated coulometric titration using a colorimetric end-point. Interference by the oxidation of organic matter is overcome by the addition of stannous chloride.

References:

Model 5030 Carbonate Carbon Apparatus Instruction Manual, Coulometrics Incorporated (1984).

Carbon Dioxide, Bicarbonate and Carbonate ions in water D513-82.

Exchangeable Cations and CEC

(LIS test names: CAESC, KKESC, MGESC, ALESC and CEC)

The capacity of clays and organic matter for the surface adsorption of large amounts of cations is due to the charge deficiency created by isomorphous substitution within the clay lattice sheets, the dissociation of hydroxyl groups from the edges of clay minerals, and the dissociation of functional groups from weak acids present in organic matter. The levels of exchangeable cations and the cation exchange capacity affects the buffering capacity of the soil, the level of plant-available nutrients, and may be important when determining the potential of the soil for acidification.

Exchangeable cations and permanent charge CEC are measured at the pH level of the soil, using 2N NaCl. A sample of air-dried less than 2 mm soil, is shaken with 2N NaCl for 4 h to allow Na^+ ions to replace ions in the exchange complex. The sample is centrifuged, filtered, and the extract analysed by atomic absorption spectrophotometry for Ca, Mg, K (and Al if the soil pH is below 5.5). Results are converted to me/100 g and the CEC is reported as the sum of the cations measured.

Aluminum is used as the measure of the acidic component. It is recognized that the CEC is underestimated for acidic soils since exchangeable H^+ is not determined. Detailed studies on acidic soils in the future will involve the measurement of exchangeable H^+. The pH dependent plus permanent charge CEC has also been measured using a buffered solution and a separate test for exchangeable H^+. However, the 2N NaCl method is better suited to large numbers of samples, provides probably the most unambiguous information on soil exchange characteristics under field conditions, and is better related to plant-available cations. A disadvantage is that Na^+ cannot be determined. However, for most Ontario soils, Na^+ contributes little to the CEC.

References:

Gillman, G. P., Bruce, R. C., Davey, B. G., Kimble, J. M., Searle, P. L. and Skjemstad, J. O. (1983). A comparison of methods used for the determination of cation exhange capacity. Comm. Soil Sci. Plant Anal. 14(11): 1005-1014.

Grove, J. H., Fowler, C. S. and M. E. Sumner. 1982. Determination of the charge character of selected acid soils. Soil Sci. Soc. Amer. J. 46: 32-38.

Osborne, V. E. 1978. Cation exchange capacity and exchangeable cations. Manual on soil sampling and methods of analysis. J. H. McKeague (ed.). Canada Soil Survey Committee on Methods of Analysis, Ottawa; 72-75.

Organically-Bound Iron and Aluminum

(LIS test names: FEEPY, ALEPY)

Sodium phyrophosphate has been found to extract humus and its complex Fe and Al salts and has little effect on amorphous or crystalline inorganic Fe and Al oxides. Pyrophosphate-extractable Fe and Al have been incorporated by the National Soil Survey Committee of Canada into their classification scheme. A 0.3 g sample of air-dried soil, ground to less than 150 µm is shaken overnight with 30 ml of 0.01 M Na-pyrophosphate. Samples are centrifuged at 20,000 rpm and the supernatant decanted and analysed for Fe, Al and Mn by atomic absorption spectrophotometry. Results are reported as percent dry weight.

References:

Bascomb, C. L. 1968. Distribution of pyrophosphate extractable iron and organic carbon in soils of various soil groups. J. Soil Sci. 19: 251-268.

McKeague, J. A. 1978. Manual on soil sampling and methods of analysis. 2nd ed. Canada Soil Survey Committee on Methods of Analysis, Ottawa; 104-105.

Oxides and Organically-Bound Forms of Iron and Aluminum

(LIS test names: FEEDI, ALEDI)

Finely divided hematite and goethite, amorphous oxides of Fe and Al and organically complexed forms of these metals can be extracted with a citrate-bicarbonate-dithionite solution. An aliquot of air-dried soil, ground to less than 150 μm, is extracted at 80°C with a sodium citrate-sodium bicarbonate-sodium dithionite solution. Sodium chloride is added to flocculate the sample. Samples are centrifuged at low speed and the supernatant decanted. Samples are extracted again, then washed twice. The final volume of extractant is diluted and Fe and Al are determined by atomic absorption spectrophotometry. Results are reported as percent dry weight.

References:

Mehra, O. P. and M. L. Jackson. 1960. Iron oxide removal from soils and clays by a dithionite-citrate system buffered with sodium bicarbonate. Clays and Clay Minerals 5: 317-327.

McKeague, J. A. and J. H. Day. 1966. Dithionite and oxalate-extractable Fe and Al as aids in differentiating various classes of soils. Can. J. Soil Sci. 46: 13-22.

Trace Metals

(LIS test names: CUUT, ZNUT, NIUT, PBUT)

Zinc, copper, nickel and lead occur in soils in amounts which vary with changing organic matter and clay content, pH, and parent material or bedrock type. These metals are determined by atomic absorption spectrophotometry after sample extraction with nitric and perchloric acids. This extraction does not remove those metals bound in silicate matrices and certain refractory oxides. The analysis, therefore, does not determine total metals, but the fraction recovered is thought to be more environmentally significant with respect to acid rain inputs to a soil system. A 0.5 g sample of air-dried soil, ground to less than 150 µm, is heated with HNO_3-$HClO_4$ in a hot-block digester for 2 h at 120°C and then at 185°C for about 5 h, until 1 ml of liquid remains. Samples are diluted to 25 ml, centrifuged, and analysed for Cu, Zn, Ni and Pb.

References:

Bock, R. 1979. Decomposition methods in analytical chemistry. International Textbook Co. Ltd. London; 212-232.

Hesse, P. R. 1971. Soil chemical analysis. John Murray Pub. London; 371-374.

Readily Soluble Aluminum

(LIS test name: ALECA)

The analytical method presently used by the Ministry removes a fraction of aluminum which strongly correlates with aluminum uptake by plants and its adverse effects on crop yields. These data may be valuable to studies on the effects of acid rain on soil. A sample of air-dried soil, ground to less than 2 mm, is shaken for 5 minutes with 0.01 M $CaCl_2$ (2:1, solution:soil ratio). The samples are centrifuged, filtered, and the extracts are acidified and analysed for aluminum by atomic absorption spectrophotometry. Manganese may also be determined in the extract: studies have found that this fraction of manganese correlates well with manganese uptake by plants.

References:

Hoyt, P. B. and M. Nyborg. 1971. Toxic metals in acid soil: I. Estimation of plant-available aluminum. Soil Sci. Soc. Amer. Proc. 35: 236-240.

Hoyt, P. B. and M. D. Webber. 1974. Rapid measurement of plant-available aluminum and manganese in acid Canadian soils. Can. J. Soil Science 54: 53-61.

Mineralogical Analysis

(LIS test name: XRD)

The mineralogical composition of a soil largely controls its chemical properties and behaviour. The weathering products, susceptibility to weathering, mechanical behaviour, buffering capacity, cation exchange capacity and available nutrient supply of the soil are some of the parameters which are a function of a soil's mineralogy.

A powder pattern of the whole sample is first prepared and X-rayed to determine the dominant minerals present in the sand and silt fraction. To determine the mineralogy of the clay fraction, the clay is separated as described in the particle size method, after pretreatment for organic matter and carbonate removal. Two aliquots of the clay fraction are removed. One aliquot is saturated with Mg^{2+} and one with K^+ to facilitate distinction between chlorite and vermiculite. The aliquots are passed through a membrane filter so that the clay remaining on the filter is oriented to permit better resolution of the clay peaks. Since most clay minerals are poorly crystalline, orienting the clay platelets improves the diffraction pattern. The clay is then transferred from the membrane filter to a glass slide by inverting the filter on the slide and applying pressure. The slides are air-dried and X-rayed. Depending on the peaks obtained, the slides are glycol-saturated and/or heat treated and X-rayed again. The mineralogy of the sample is determined through interpretation of the powder pattern and four X-ray traces of the clay fraction.

References:

Brown, G. 1961. The X-ray identification and crystal structure of clay minerals. Mineralogical Society of London.

Carroll, D. 1970. Clay minerals: A guide to their X-ray diffraction. Geological Society of America. Special Paper 126. Colorado, U. S. A., p. 80.

Whittig, L. D. 1965. X-ray diffraction techniques for mineralogical identification and mineralogical composition. Methods of soil analysis. Part 1. C. A. Black et. al. (eds.). Agronomy Monograph No. 9, American Society of Agronomy, Madison, Wisconsin; 671-698.

Soil-Acid Equilibration (Buffering Curve)

(LIS test name: BUFCRV)

This method was devised specifically for the APIOS Terrestrial Effects program to examine the ability of a soil to buffer inputs of sulphuric acid. Nine, 10 g aliquots of air-dried less than 2 mm soil, are weighed into separate beakers. Twenty ml of distilled water is added to the first beaker and 20 ml of varying concentrations of sulphuric acid are added to the remaining beakers (meq concentrations vary from 0.02 meq for 20 ml of 0.001 N H_2SO_4, to 1 meq for 20 ml of 0.05N H_2SO_4). Samples are stirred for 30 minutes, left to equilibrate for 1 h, and pH is then determined. Results are plotted (pH vs meq concentration) with each aliquot representing a point on the curve. This method avoids dilution problems associated with a regular titration. The equilibrating solution can be altered, if desired, to more accurately reflect the ion concentrations of acid precipitation in Ontario.

Soluble Sulphate

(LIS test name: SSO4EW)

Sulphate is an important ion in the Terrestrial Effects Study because it is added to the soil by the precipitation itself as well as through mineralization of organic sulphur compounds. Water-soluble, plant-available sulphate is extracted by shaking a 5 g sample of air-dried less than 2 mm soil, with 25 mℓ of distilled water. Although saturated pastes and 2:1 solution:soil ratio have also been used, the 5:1 ratio was chosen as it accomodates organic samples and yields enough sample to permit membrane filtration. After being shaken for 1 h, samples are centrifuged and filtered through a 0.45 µm membrane filter. Extracts are analysed for SO_4 by ion chromatography.

References:

Ballantyne, A. K. 1978. Soluble salts. Manual on soil sampling and methods of analysis. J. A. McKeague (ed.) Canada Soil Survey Committee on Methods of Analysis, Ottawa; 71.

Adsorbed Sulphate

(LIS test name: SSO4AD)

Adsorbed SO_4 is measured by shaking a 5 g sample of air dried soil (<2 mm size fraction) with 25 mℓ of NaH_2PO_4 solution containing 1000 µg/mℓ P. Samples are centrifuged and filtered through a 0.45 µm membrane filter. Extracts are analysed for SO_4 by ion chromatography using a KHP eluant. For highly organic samples a 1:20 soil:solution ratio is used for extraction.

References:

Ensminger, L. E. (1954). Some factors affecting the adsorption of sulphate by Alabama soils. Soil Sci. Soc. Amer. Proc. 18:259-264.

Johnson, D. W., Henderson, G. S. and Todd, D. E. (1981). Evidence of modern accumulations of adsorbed sulfate in an east Tennessee forested Utisol. Soil Science 132(6):422-426.

Total Nitrogen

(LIS test name: NNTKUR)

Nitrogen compounds are converted to simple inorganic forms (ammonium ions) by sample dissolution in sulphuric acid and persulphate. A sample size of 0.08 g to 0.4 g is required, air-dried and ground to less than 150 μm. The potassium persulphate increases the boiling point and provides an oxidizing environment. Iron and calcium interference is removed by filtration, after making the solution strongly basic. Ammonia is analysed colorimetrically in the digestate.

References:

Ontario Ministry of the Environment. 1983. Handbook of Analytical Methods for Environmental Samples. Laboratory Services Branch. pp. 246.

Organic Carbon

(LIS test name: ORGC)

Beginning in 1984/85, organic carbon is reported as the difference between total carbon, as measured by a LECO CR-12 carbon analyser, and inorganic carbon (see method for carbonates). The method correlates well with the previous method which involved oxidation with potassium dichromate and sulphuric acid and the colorimetric measurement of Cr^{3+}.

In the LECO CR-12 total carbon analyser system, oxygen is purged through the system and samples are combusted at 1370°C oxidizing carbon to CO_2. The CO_2 is collected, passed through several traps to remove moisture and then measured by an infrared detector.

References:

LECO Instruments manual for the CR-12 system.

VEGETATION SAMPLES

Total Chlorine and Sulphur

(LIS test names: CLUT, SSUT)

These nutrients are all measured on a vegetation pellet by X-ray fluorescence spectrometry. A sample of dried, ground vegetation is weighed into a clean plastic vial. Powdered wax is added to the vial, the vial is capped, then shaken until a homogenous mixture is obtained. The mixture is transferred to a die and pelletized with a pressure of 25 tons. Samples are analysed for total S, and Cl by X-ray fluorescence spectrometry.

References:

Judge, R. H. and J. A. Pimenta. 1980. The analysis of plant material by X-ray fluorescene spectrometry, Part 1: chlorine, sulphur, phosphorus, calcium, potassium, silicon. Ministry of the Environment, Laboratory Services Branch, Rexdale, Ontario.

Norrish, K. and J. T. Hutton. 1977. Plant analysis by X-ray spectrometry: low atomic number elements, sodium to calcium. X-ray Spectrometry. 6(1): 6.

Total Magnesium, Sodium, Aluminum, Manganese, Iron, Copper, Zinc, Molybdenum, Nickel, Lead, Cadmium, Calcium, Titanium, Potassium, Boron, Cobalt, Chromium, Vanadium, Silver.

(LIS test names: MGUT, NAUT, ALUT, MNUT, FEUT, CUUT, ZNUT, MOUT, NIUT, PBUT, CDUT, CAUT, TIUT, KKUT, BBUT, COUT, CRUT, VVUT, AGUT)

A sample of dried, ground vegetation is weighed into a crucible and ashed in a muffle furnace at a temperature below 150°C. The temperature is then raised to 250°C for 1 h, and to 500°C for 3 h. The sample is then digested in aqua regia at moderate heat. The digestate is diluted with distilled water and analysed for the above elements by ICP. For Ti and Ag analysis, a HNO_3-H_2SO_4 digestion is used instead of aqua regia.

References:

Ontario Ministry of the Environment. Handbook of analytical methods for the analysis of environmental samples (1983). Laboratory Services Branch.

Total Nitrogen and Phosphorus

(LIS test name: PPUT, NNTKUR)

Samples are digested in sulphuric acid and potassium persulphate. A sample of approximately 0.02 g, dried and ground to <40 mesh, is required. The potassium persulphate increases the boiling point and provides an oxidizing environment. Iron and calcium interference is removed by filtration after making the solution strongly basic. Ammonium and phosphorus are analysed colorimetrically in the digestate.

References:

Ontario Ministry of the Environment. 1983. Handbook of Analytical Methods for Environmental Samples. Laboratory Services Branch. pp. 246.

Fluoride

(LIS test name: FFUT)

A dried and ground vegetation or soil sample is extracted with 0.1 N perchloric acid in a water bath at 80°C. A buffer is added to soil samples and all samples are shaken. Fluoride is determined in the soil or vegetation suspension by ion selective electrode.

References:

Nicholson, K. and Duff, E. J. (1981). Fluoride determination in water: an optimum buffer system for use with the fluoride-selective electrode. Analytical Letters 14 (A7):493-517

Vijan, P. and Alder, B. (1984). Fluoride analysis in vegetation by ion selective electrode. American Laboratory.

Villa, A. E. (1979). Rapid method for determing fluoride in vegetation using ion-selective electrode. Analyst 104:545-551.

Total Mercury

(LIS test name: HGUT)

Vegetation samples are digested in 4:1 H_2SO_4:HNO_3, diluted to 25 mℓ with distilled water and analysed by flameless atomic absorption spectrophotometry. More detailed information is available in the Ministry's "Handbook of Analytical Methods for Environmental Samples". Laboratory Services Branch.

References:

Ontario Ministry of the Environment. 1983. Handbook of Analytical Methods for Environmental Samples. Laboratory Services Branch.

Starch Analysis

Starch is determined in vegetation samples by the University of Guelph under the direction of Dr. B. Grodzinski. Samples are submitted after three washings in 80% ethanol and drying overnight. About 20 mg of dried sample is submitted in test tubes. Samples are then rehydrated in 0.1 m sodium acetate (pH 4.5), 1 mℓ amyloglucosidose enzyme (10 mg/mℓ) is added and samples are shaken at 50°C for 4 hours. Serum separators are used to remove particulate and an assay for glucose is done using anthrone reagent. Further information can be obtained from D. McLaughlin, Phytotoxicology Section, Air Resources Branch, Ministry of the Environment.

PIXE Analysis (Proton Induced X-ray Emission)

Tree core samples are analysed for various elements under the direction of D. McArthur, Physics Department, Queen's University, Kingston, Ontario. Further information regarding PIXE analysis can be obtained from D. McLaughlin, Phytotoxicology Section, Air Resources Branch, Ministry of the Environment.

APPENDIX

SITE DESCRIPTION FORM

1. SITE NUMBER: ______________________________

2. FOREST REGION: ____________ Deciduous ______________
Great Lakes St. Lawrence ______________

3. FOREST SECTION: ______________________________

4. COUNTY OR DISTRICT/TOWNSHIP: ____________/ ____________

5. LOT/CONCESSION: ______________/ ______________

6. LAND TENURE: PUBLIC: ____________ PRIVATE: ____________
FEDERAL: ____________________ NAME: ______________
PROVINCIAL: __________________ ADDRESS: ____________
MUNICIPAL: __________________ ______________
______________________________________ park/C.A.

7. NTS 1:50,000 map number: __________________________

8. UTM: ZONE: ________ EASTING: ________ NORTHING: _______

9. FRI MAP NUMBER: ______________________________

10. NEAREST AIR MONITORING SITE: AES: __________ MOE: _______
PRIVATE: ________________

11. NEAREST APIOS SOIL BASELINE PIT: PIT NUMBER: ____________

12. NEAREST POPULATION CENTRE:
____________________: __________ km ______________

SURVEY I. D.

DATE: ______________________

Soil/Site
PLOT DESCRIPTION FORM

PLOT NO.: ______________________
SURVEYORS: ______________________

1. Location on the Slope:

C	Crest	U	Upper
M	Middle	L	Lower
T	Toe	D	Depression
F	Flatland		

2. Aspect (average condition for the plot):

3. Rock Outcrops (indicate % surface coverage to nearest 10%):

4. Surface Stoniness:

FREQUENCY		SIZE	
N	non-stony	S	small
S	slightly	M	medium
M	moderately	L	large
V	very	B	boulders

5. Soils Data

SOIL PROFILE DESCRIPTION

Horizon	Thickness	Texture	G/g

Depth to Carbonates (cm):

Depth Organic Matter (avg. of 5 samples):

Moisture Regime:

0 0 1 2 3 4 5 6

pH Class (at 20 cm depth):

1 2 3 4 5 6

6. Soil Sample Number:

NOTES:

FORESTRY

SURVEY I. D.

DATE: ____________________
PLOT NO.: ____________________
SURVEYORS: ____________________

PLOT DESCRIPTION FORM

7. Basal Area:

Species	No. of Trees					Species Totals
	1	2	3	4	5	
TOTAL TREES						

Mean Basal Area (m^2/hectare):

8. Species Composition (percent classes):

9. Map (draw on back of sheet):

10. NOTES:

CROWN CONDITION CLASSIFICATION SYSTEM

RATING	DESCRIPTION
1	Near perfect specimen tree.
2	Forest tree with high quality crown, and with no dead branches occurring above the base of the crown.
3	Tree crown in good condition, with 1 or 2 dead branches above the base of the crown.
4	Tree crown in fair to moderate condition, with at least three dead branches and up to 25% of the crown dead.
5	Between 25 and 50 percent of the crown dead.
6	Between 50 to 75 percent of crown dead.
7	Between 75 to 90 percent of crown dead.
8	Over 90 percent of crown dead, some branches retaining foliage.
9	Most of crown dead, little foliage left on any one branch.
10	Entire crown dead.

NOTE: The base of the crown is defined as the lowest live branch on the tree, not including epicormic branches or sprouts.

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